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KERNFORSCHUNGSZENTRUM

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Institute for Materials and Solids Research *)

Creep Rupture Strength and Creep Behavior of Vanadium/Titanium and Vanadium/Titanium/ Niobium Alloys

H. Böhm, M. Schirra

OFFICIAL REPORT
USAEC-EURATOM
Fast Reactor Exchange

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Gesellschaft für Kernforschung mbH, Karlsruhe

1. Introduction

Increasing interest has recently been shown in vanadium alloys since a number of properties render them suitable as canning materials for fast reactors. The requirements imposed on a material used for these purposes are high creep rupture strength in the range 600 - 800°C, the minimum possible damage due to neutron irradiation at low absorption cross sections for fast neutrons, adequate resistance with respect to the coolant (sodium) and the fuel (uranium oxide or uranium carbide) and good workability. $] \longrightarrow p. 4$

As compared with the austenitic steels planned to be used as canning materials in fast sodium-cooled breeder reactors, the vanadium alloys display a number of advantages justifying the interest in them. In the temperature range considered, for example, they have a much higher creep rupture strength (Ref. 1) than the high-temperature austenitic steels and, in particular, show no signs of high-temperature embrittlement due to neutron irradiation (Ref. 2). If metal fuel is used (uranium/plutonium/fissium alloys), the vanadium alloys are also clearly superior to the austenitic steels with regard to their compatibility with the fuel (Ref. 3). The corrosion behavior of the majority of vanadium alloys with respect to sodium is less good (Refs. 4 and 5), but promising development work is being conducted in this field. For helium-cooled fast reactors, vanadium alloys already constitute a suitable canning material since the corrosion problem largely no longer arises.

Important alloying elements for vanadium alloys
have found to be titanium and niobium (increase
in strength) as well as aluminum and chromium
(improvement of resistance to corrosion).
Despite the large number of alloys already
tested, although these experiments largely
consisted in screening tests, there is a
widespread lack of systematic studies on the
creep rupture strength and creep behavior
of vanadium alloys, especially with regard
to the long-time behavior.

Results concerning the creep rupture behavior
of vanadium alloys have been elaborated by
Rostoker and co-workers (Refs. 6 and 7) for
vanadium alloys containing 40 - 60 % of titanium
and additions of aluminum, chromium and carbon,
while van Thyne and co-workers (Refs. 8 - 11)
have obtained results for vanadium alloys
with titanium contents of up to 20 % and niobium
contents of 20 and 60 %. Studies were also
carried out on the influence of carbon, nitrogen
and boron with the aim of improving the heat
resistance of the alloys by a finely distributed
separation of solid phases. The strength
values listed in the works mentioned mainly
relate to fairly short residence times.

W. Pollack and co-workers (Ref. 12) report
on a comprehensive program for the development
of creep-resistant vanadium alloys. The aim
was to increase the creep strength by the
combination of solid solution hardening (via W,
Mo, Cr, Ta, Nb, Ni, Fe, Ti, Al) and dispersion
hardening (by the formation of carbides or
nitrides of titanium and zirkonium). Alloys
containing 8.5 % Cr, 9.9 % Ta, 1.24 % Zr and
0.05 % C and 9.1 % Cr, 3.3 % Fe, 1.3 % Zr and
0.05 % C were found to be particularly suitable.

The first results obtained by us from a systematic

study on the creep rupture strength and creep behavior of vanadium/titanium and vanadium/tinanium/niobium alloys were recently reported on (Ref. 1). The subsequent results are reported in the present work and a general account given of the creep rupture strength and creep behavior of this group of alloys.

2. Test Material

The studies were carried out on the alloys listed in Table 1. Together with vanadium, the alloy elements titanium and niobium form homogeneous solid solutions in the concentration ranges given. Owing to the interstitial impurities oxygen, nitrogen and carbon, the average content of which was about 600 ppm C, 1000 ppm O_2 and 400 ppm N_2, however, finely distributed oxide or carbide precipitations appear in the alloys.

The alloys were made by the Metallgesellschaft from the pure components, swaged into rods 8.5 to 10 mm in diameter and finally vacuum annealed for one hour at 900 - 950°C. The starting textures of the alloys studied are given in Figs. 1a - d.

The metallographic examination shows that annealing did not lead to a completely recrystallized texture in all the alloys, the alloys with a low titanium and a high niobium content displaying no or hardly any signs of incipient recrystallization. As the hardness measurements showed, however, a virtually complete removal of work hardening occurred in all of them. It is known that both in the case of pure vanadium (Ref. 13) and vanadium alloys (Ref. 14) large-scale recovery of the

mechanical properties is possible without recrystallization. The influence of the titanium on the recrystallization behavior is very marked, as can be seen from Figs. 1a - d (Ref. 1). Afterwards the recrystallization temperature drops with increasing titanium content above $\approx$ 3 % Ti. However, for the purposes of the studies, it appeared desirable to carry out the same annealing in all the alloys. Generally speaking it was found that the secondary creep rate $\dot{\varepsilon}$ is somewhat lower in alloys in the recrystallized state than after stress relief annealing. However, the heat treatment cannot influence the creep behavior solely via recovery and recrystallization, but also by changing the quantity and distribution of the carbide or oxide precipitations, which are clearly visible in the micrographs and have also been observed and identified by other authors (Ref. 3). The influence of the cold work and heat treatment on the creep rupture properties is to be discussed in a subsequent publication.

3. Experimental Procedure

The creep rupture and creep tests were carried out in vacuum creep test rigs at a pressure of $\approx$ 1 x 10^{-5} torr (Fig. 2). Threaded samples 5 mm in diameter and 25 mm long were selected as the samples. In order to achieve a constant temperature over the entire length of the sample, it is surrounded with a shielding sheath. In long-time experiments ($>$ 1000 h) the space between the sample and the sheath was filled with titanium swarf in order to preclude the possibility of surface oxidation by gettering. The test temperature was maintained constant to $\pm$ 2°C by three individually adjustable heating zones.

The sample elongation was measured continually
during the test via the upper attaching
bolts by means of gages or inductive recorders.

4. Test Results and Evaluation

4.1 Creep Rupture Strength → / ¢. 8

The studies aimed at determining the creep
rupture strength of the alloys listed were
carried out mainly at 650°C. In order
also to obtain data on the long-time behavior
or the alloys, the experiments were extended
to residence times of up to > 10,000 hours.

The results of the studies, including the
values published in a previous work, are
listed in Figs. 3 - 6. Each figure contains
the creep rupture curves for alloys with a
constant niobium content and the titanium
content as a parameter. The values confirm
the results obtained previously on a much
smaller number of alloys (Ref. 1), according
to which the short-time strength at 650°C
increases for rising titanium content, but
the long-time strength drops.

A general picture of the creep rupture behavior
of the V-Ti and V-Ti-Nb alloys is given in
Figs. 7 - 9, which show specially the stresses
for residence times of 100, 1000 and 10,000 hours.
As can be seen from Fig. 7, the 100 hour creep
rupture strength rises for increasing niobium
and decreasing titanium content and in the
concentration range studied reaches a maximum
for 15 % of niobium and 3 % of titanium
with a marked drop for lower titanium contents.
The 1000 hour creep rupture strength (Fig. 8)
shows the same dependence on the niobium
content, but with regard to the titanium content

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the strength maximum is extended to the range from 1 to 3 %. The values for $\sigma_{B/10000}$ (Fig. 9), which were extrapolated on the basis of the available results or interpolated from long-time studies now in progress ($>$10,000 hours), indicate that the strength peak is further displaced towards lower titanium contents ($\approx$ 1 %) and the influence of the niobium content on the strength constantly decreases, at least in alloys with low titanium contents.

In addition to the studies at 650°C already mentioned, creep rupture values were also obtained for certain alloys at higher temperatures in order to provide an indication of the temperature-dependence of the creep rupture strength. For the binary alloys V-5Ti and V-20Ti the measured values are plotted in Figs. 10 and 11 together with the values for 650°C. It will be seen that, for decreasing titanium content, not only is the long-time behavior at 650°C improved, but also the creep rupture strength at higher temperatures. On ternary V-Ti-Nb alloys, only individual tests at temperatures of up to 920°C were carried out. With the exception of those for V-5Ti, the results are shown in Fig. 12. For purposes of extrapolation, the creep rupture strength values for certain alloys are plotted here versus the Larson-Miller parameter $P = T(C + \log t_B)$, with a value of 15 for the constant C.

As the results have shown, the V-Ti-Nb alloys can be regarded as decidedly temperature-resistant materials. The long-time behavior of the best V-Ti-Nb alloys corresponds to that of the sophisticated precipitation-hardening nickel alloys. Moreover, by the addition of small quantities of silicon it is possible to increase the creep rupture strength further and to achieve $\sigma_{B/1000}$ values of $\approx$ 70 kp/mm^2 at 650°C. As a result these

alloys are superior to all known materials
with regard to their creep rupture behavior.

4.2 Creep Behavior $\rightarrow p.12$

In order to obtain precise data on the creep
behavior of the material it is necessary to
determine the stress and temperature dependence
of the secondary creep rate.

In many cases the stress and temperature
dependence of the secondary creep rate $\dot{\varepsilon}$
can be satisfactorily described for low
stresses by the formula (Refs. 15 and 16)

$$\dot{\varepsilon} = A \left(\frac{G}{E}\right)^n e^{-Q_K/RT}$$

where Q_K is the activation energy of creep,
n the so-called stress exponent, E the modulus
of elasticity and A a structure-dependent
factor. The activation energy Q_K is in many
cases equal to the activation energy Q_D of the
diffusion.

The activation energy which is normally determined
from measurements of the creep rate at different
temperatures on the basis of the equation

$$\dot{Q_K} = R \frac{\Delta \ln \dot{\varepsilon}}{\Delta \frac{1}{T}}$$

deviates somewhat from the true activation
energy Q_K, since no account is taken of the
temperature-dependence of the modulus of elasticity.

The above formulae give the activation energies
on the assumption that the stress exponent n
is temperature-independent, which is to be
expected in accordance with the theories
of secondary creep, provided that no change
occurs in the creep mechanism.

In a previous publication (Ref. 1), values
for the activation energy of creep and of
the stress exponent for various vanadium
alloys for the temperature range 650 - 750°C
were reported and briefly discussed. A noteworthy
feature was the marked dependence of the
activation energy on the titanium content
as well as the values measured for high
titanium contents were well above the values
for the activation energy of diffusion.
The very low activation energy found in
vanadium alloys with $\approx$ 3 % of titanium,
in conjunction with a relatively high stress
exponent n, was found to be due to the fact
that the creep processes in the temperature
range studied are not diffusion-controlled.
The results of this first investigation
provided the impetus for carrying out more
comprehensive measurements on a large number
of binary V-Ti and ternary V-Ti-Nb alloys,
the temperature range studied being extended
to about $\approx$ 900°C.

The activation energy was determined in the
familiar manner by measuring the creep rate $\dot{\varepsilon}$
of a sample under constant stress at different
temperatures. The creep time at each
temperature was about 70 - 100 hours before
the temperature was rapidly raised by about
10°C in each case under stress. In a number
of samples the temperature was alternately
raised and lowered in order to detect possible
changes in the creep rate as a result of
texture modifications. In no case, however,
was the creep rate found to have any influence
which extended beyond the normal scatter range.

Figs. 13 - 17 give the temperature-dependence
of the creep rate for the alloys whose creep
behavior was studied over a wide temperature
range. Since it is not possible to conduct
the tests over the entire temperature range
with one single stress, the creep rate curve
shown in Figs. 13 - 17 is made up of several
different sections which are displaced parallel
in the direction of the ordinates and cover
the creep rate range of 10^{-5} - 10^{-4}/hour
for the particular stress. The activation
energies of the creep given in the figures
are thus valid for comparable creep conditions
($10^{-5} < \dot{\varepsilon} < 10^{-4}$/hour). As can be seen,
the stress has no influence on the activation
energy within the creep rate range taken as the
basis.

It can be seen from the figures that the creep
processes in the alloys containing 10 % and
20 % of titanium take place at a much higher
activation energy at temperatures of below
$\approx$ 700 - 720°C than at higher temperatures.
A temperature-independent activation energy
is found at 5 % of titanium, while for lower
titanium contents the activation energy is
lower at temperatures below 700°C than at higher
temperatures.

In Fig. 18 the uncorrected activation energies
obtained from measurements between 650 and
700°C are plotted for all the binary and ternary
vanadium alloys studied as a function of the
titanium content, the numerical values indicating
the stresses at which the tests were carried
out. The measurements confirm the results
already published (Ref. 1) concerning the
influence of the titanium on the activation
energy of the creep in this temperature
range and show again that the niobium content
has no noticeable influence. A completely
different dependence on the titanium content
is shown by the activation energy at temperatures

of above 750°C (Fig. 19). Here there is only
a very slight constant increase in the activation
energy with the titanium content. The high
activation energy of creep observed for titanium
contents of 10 % and above is therefore limited
to a narrow temperature range, the creep
behavior at temperatures below 650°C still
having to be determined.

The previous findings were confirmed with
regard to the stress dependence of the creep
rate. Figs. 20 - 23 give the creep rate $\dot{\varepsilon}$ as
a function of the stress for 650°C (and in
some cases 700 and 800°C) for all the alloys
studied. It was found that the dependence
$\dot{\varepsilon} = k \cdot \sigma^n$ is satisfactorily fulfilled in
all cases. The stress exponent n obtained
from Figs. 20 - 23 is given in Fig. 24 for
the binary and ternary alloys as a function
of the titanium content. Here the stress
exponent shows a maximum for the test temperature
of 650°C at low titanium contents, while
the value for alloys with a higher titanium
content is in the range to be expected with
diffusion-controlled creep processes. As
far as can be seen from the few results available,
the maximum decreases for rising temperature.
A reduction in the stress exponent for increasing
temperature also occurs at higher titanium
contents as can be seen from Fig. 24. Table 2
gives the n-values with the corresponding
K-values for all the vanadium alloys studied.

While knowledge of the stress dependence and
temperature dependence of the secondary creep
rate is of decisive importance for understanding
and explaining the processes which occur during
creep, the creep limits provide an important
characteristic for practical application which
is determined by the creep behavior. For
all the alloys studied, therefore, the 1 %
and 2 % creep limits were determined from
the creep curves, these being given in Figs. 25

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- 29.   Owing to the indirect elongation measurement via the attaching bolts, there is in some cases marked scatter of the 1 % limits in particular.

In order to be able to detect the influence of the titanium and niobium content on the ratio creep limit/creep rupture strength, Fig. 30 given the ratio $\sigma_{2/100}/\sigma_{B/100}$ and $\sigma_{2/1000}/\sigma_{B/1000}$ as a function of the titanium content. There is a clear reduction in the ratio for increasing titanium contents, but no marked influence of the niobium is to be observed.

4.3 Breaking Elongation

The breaking elongation measurements indicate that here, too, the titanium has a marked influence.   The breaking elongation values measured during the creep rupture test at 650°C (Figs. 31 and 32) point to a clear pattern despite the scatter, which is in some cases severe.   Whereas, for a titanium content of 3 %, the breaking elongation depends only very slightly on the holding time, it increases with the time for higher concentrations.

As is shown in the following section, the relatively marked increase in the breaking elongation with the holding time for higher titanium contents is related to incipient recrystallization.   The alloys containing 3 and 5 % of titanium, on the other hand, show no signs of recrystallization even after very long holding times at 650°C, so that the dependence of the breaking elongation on the titanium content is doubtless connected with the influence which the titanium has upon the recovery and recrystallization behavior.

4.4 Metallographic studies *)

The metallographic studies were used to obtain
more detailed data on the rupture behavior
of the alloys and on the recrystallization
phenomena which occur during the creep rupture
test. The micrographs of the fracture zone
of alloys V-2Ti-15Nb and V-5Ti given in Fig. 33
are characteristic of the rupture behavior.
According to them the alloys studied show
a transcrystalline fracture. At higher
temperatures also the fracture is transcrystalline
despite isolated intercrystalline incipient
cracks (Fig. 34).

Knowledge of the rupture behavior of vanadium
alloys is important with a view to their
use as canning material in reactors, since
the high-temperature embrittlement due to
neutron irradiation rises as the tendency
of the alloys towards intercrystalline fracture
increases. In accordance with the rupture
behavior results obtained, it was shown on a
number of vanadium alloys (Ref. 2) that
no high-temperature enbrittlement occurs
in the temperature range of 600 - 750°C concerned.

It has already been pointed out that the
recrystallization behavior of the vanadium
alloys is to a large extent governed by
the titanium content. While the recrystallization
of cold-worked samples takes place between
850°C (for ≈ 20 % titanium and 50 % cold work)
and 1000°C (for 3 % titanium and similar
cold work), depending on the titanium concentration
and cold work (Ref. 14), recrystallization
processes occur at much lower temperatures
during this creep test. In addition to a

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*) The comprehensive metallographic studies were
   carried out by Mrs. Junge.

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complete recrystallization with a generally
speaking very fine-grained texture, one
also observes serrated grain boundaries,
which were also detected in other alloys
(Refs. 17 - 19) during a creep study.

The metallographic studies conducted on
numerous samples indicate that the occurrence
of serrated grain boundaries can be regarded
as a precursor of complete recrystallization,
i.e., that it occurs at somewhat lower tempera-
tures or lower deformations than the primary
recrystallization. As Fig. 35 shows for a
V-3Ti-20Nb alloy, for example, almost complete
recrystallization is observed in the rupture
zone. In the range outside necking, i.e.,
for lower deformation, predominantly serrated
grain boundaries are found, while only the
starting texture is found in the top section
of the sample. Such grain boundaries occur
at temperatures as low as 650°C in long-time
creep tests even in alloys with a low titanium
content, which are marked by a relatively
high recrystallization temperature. The
formation of new grains during primary
recrystallization takes place both in the
grain and at the severely serrated grain
boundaries (Fig. 35).

Complete recrystallization during the creep
rupture test is observed in alloys with
titanium content of 20 % between 750 and
800°C (depending on the stress applied)
and in alloys containing 3 - 5 % titanium
at $\approx$ 850°C, i.e., the recrystallization
temperature in the creep test is well below
that determined on cold-worked samples.

Discussion of Test Results

The results obtained show clearly the marked
influence which the titanium content of the

vanadium alloys has on the holding time t_B and the secondary creep rate $\dot{\varepsilon}$. For the discussion of the results it is important to know whether the dependence of the holding time on the titanium content is due solely to the dependence of the creep rate on the titanium content or whether the titanium also changes the holding time by influencing the rupture processes. The correlation between time and creep rate can generally be satisfactorily described by the equation

$$log\ t_B + m\ log\ \dot{\varepsilon} = K$$

where m (usually $\approx$ 1) and K are alloy-dependent quantities (Ref. 20). If the titanium content influences the time only via the creep rate, the quantities m and K should be independent of the titanium content, i.e., the values of $\dot{\varepsilon}$ and t_B should lie on one curve for all vanadium alloys. If allowances are made for the fairly marked scatter in the values of $\dot{\varepsilon}$ and t_B, it can be seen (Fig. 36) that the values for the various vanadium alloys lie within a scatter band. A precise analysis of the groups of values indicates that the holding time is reduced somewhat more slowly for increasing titanium content than should be the case if the holding time reduction were solely determined by the creep rate change. However, the difference is so slight that the holding time can be regarded as a function of the creep rate to a good approximation and hence the main stress in the discussions can be placed in the interpretation of the creep processes. If the activation energy and the stress exponent are first regarded as the quantities characterizing the creep process, then the values measured for the temperature range above 750°C are readily understandable.

The creep activation energies determined
for this temperature range are in good agreement
with those for the diffusion in pure vanadium
and vanadium/titanium alloys, so it can be
assumed with certainty that the creep at
these temperatures is determined by the
diffusion of vanadium and titanium and/or
niobium atoms. A decision as to which diffusion
process is the main one cannot be taken
on the basis of the measured activation
energies alone*), since no values are available
for the diffusion of niobium in vanadium/titanium
alloys. It is advisable to regard the climbing
of dislocations as the process which determines
the creep rate. The few stress exponent
values measured for the temperature range
above 750°C are in agreement with this.
This provides further confirmation of the
theory that the creep processes in body-centered
cubic alloys are not fundamentally different
from those in metals with high-density spherical
systems, as Sherby (Ref. 21) and Vandervoort
(Ref. 22) have already shown.

The creep processes at lower temperatures
are much more complicated. The low activation
energies and high stress exponents measured
in the 650°C range for alloys with titanium
contents of under 5 % suggest that the creep
processes in this concentration and temperature
range are not controlled by the diffusion
of V, Ti or Nb atoms. Dislocation climbing
can therefore not be regarded as a process
which determines the rate.

On the basis of the available results it is
not possible to decide which process determines

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*) The values Q_K for the activation energy, which
were corrected with allowances for the tempera-
ture-dependence of the modulus of elasticity,
are only slightly below the indicated values of
Q_K' , so that no useful conclusions can be
drawn from the corrected values either.

the creep is this concentration and temperature
range. On account of the in some cases
very low activation energies, which are
within the range of values for the diffusion
of interstitial impurities such as oxygen,
nitrogen and carbon, it might at first
be thought that dislocation blockage occurs
owing to the elements listed, the creep
process then being determined by the diffusion
of the blocking atoms (Refs. 23 and 24).
Apart from the fact that a much lower
stress exponent would be to be expected
in this case (Ref. 23), alloys have shown
(Ref. 25) that no marked yield point occurs
for titanium contents of below 5 %, i.e.,
the mechanism indicated.

While, in the case of low titanium alloys,
it can hence be assumed that the creep
behavior at 650°C is not diffusion-controlled,
a diffusion-controlled creep process is
likely at these temperatures in alloys
with higher titanium contents (>10 %).

This is borne out if only by the fact
that in these alloys marked recovery processes
occur at the temperature under consideration,
and, in the case of long-time creep tests,
even incipient recrystallization is observed.
The very high activation energy in this
temperature range, however, does not permit
such a simple explanation of the creep
process as at higher temperatures.

It might first be assumed that the recrystalliza-
tion which commences during creep simulates
the very high activation energy, which,
for example, is assumed to be the case
with regard to the very high activation
energy of creep in aluminum monocrystals.
(Ref. 26). In this case one would expect a
corresponding rise in the activation energy

even in alloys with lower titanium contents,
although at higher temperatures. However,
since none of the alloys studied with
titanium contents of less than 10 % showed
this increase in the activation energy,
not even at temperatures at which a recrystalliza-
tion occurs during the experiment, it can
be broadly speaking assumed that the high
value is not simulated by incipient recrystalliza-
tion.

It is more likely that the causes are to be
sought in an interaction between the oxygen
atoms and the titanium atoms and dislocations.
A factor militating in favor of this is
that in the alloys studied only titanium, which
possesses pronounced interactions with
the interstitial impurities, exercises
a marked influence upon the creep activation
energy. Moreover, it is known (Ref. 25)
that, contrary to what might be expected,
a pronounced yield point due to interactions
between interstitial impurities and dislocations
occurs with high titanium contents, but
not at low contents, so that the titanium
probably does not completely react with
the interstitially dissolved elements.
The high activation energy may then possibly
be ascribed to the dislocation blockage
or the removal of this blockage. This
assumption is borne out by the results
of hot tensile tests on alloys containing
20 % of titanium at higher temperatures.
It was found that a marked yield point
occurs in this alloy only up to about
650°C, i.e., above this temperature there
is no more dislocation blockage due to
the interstitial impurities, while alloys
containing 5 % of titanium (no yield point
effect is observed below 5 %) display a
marked yield point even at 850°C.

Similar behavior with regard to the activation
energy of creep is shown by an aluminum

alloy containing 3.2 % of Mg, in which a
marked rise in the activation energy was
recorded in a narrow temperature range
(Ref. 27). Here, too, dislocation blockage
phenomena are regarded as a possible cause
of the extreme activation energy value, in
accordance with Cottrell's mechanism.

The available results, particularly those
concerning the concentration-dependence
of the creep parameters, provide initial
indications of the possible processes involved,
as has been shown, but it is not yet possible
to obtain a precise and detailed explanation
of the unexpected dependency of the creep
rate and residence time on the titanium
content. It would appear to be necessary
here to study the influence of the content
of interstitial impurities, which according
to the remarks made above are of importance
for the creep behavior. The fact that alloys
made from different starting materials show
no abnormal scatter suggests that there
is noticeable relation between the creep
behavior and the concentration of these
elements for relatively high oxygen, nitrogen
and carbon contents (together $> \approx 2000$ ppm).
However, tests are now being conducted to
determine how the creep behavior changes
for much lower contents of these elements.

Very little data are available either on
the almost complete recovery of the vanadium
alloys without recrystallization, which
doubtless had a decisive influence upon
the creep behavior. This is likely to be
the reason for the unexpected creep behavior
of cold-worked samples, in which breaking
elongation values of > 150 % were observed
in the creep test. (Ref. 28). It is not
yet possible to say to what extent the recovery
processes are related to the content of
interstitial impurities.

At creep temperatures of above 650 – 700°C
(depending on the titanium content) the
pure recovery processes are accompanied by
grain boundary migration and recrystallization.
The formation of serrated grain boundaries
is particularly striking.

Since this form of grain boundary is not
observed in the recrystallization of cold-worked
vanadium samples but only during creep, its
occurrence must be closely related to creep
deformation. The marked increase in the
grain boundary surface and hence in the boundary
surface energy as a result of the formation
of serrated grain boundaries indicates that
there must be a strong driving force for
grain boundary migration, which, on the basis
of the average radius of curvature of the

teeth in the grain boundaries, is found to be
$\approx 10^7$ dyn/cm^2, a grain boundary energy
of 1000 erg/cm^2 being assumed.

The fact that serrated grain boundaries only
occur during creep, but not during recrystalliza-
tion, can have various reasons. For example,
deformation during creep at higher temperatures
produces a different dislocation arrangement
than at low temperatures and hence influences
the grain boundary migration during recrystalliz-
ation. Seen from this angle, the results
obtained hitherto concerning the occurrence
of serrated grain boundaries are also explained.
It is assumed that a very pronounced sub-grain
formation occurs in the region of the grain
boundaries during creep in the event of
grain boundary slip, where the size of the
sub-grains on both sides of the grain boundary
can differ considerably. In this case grain
boundary migration in the direction of the
small sub-grains would occur (strain-induced
grain boundary movement), which may lead
to the formation of grain boundaries described.

This mechanism can not be ruled out in the
vanadium alloys either, especially since
the marked recovery of the mechanical properties
without recrystallization point to a pronounced
sub-grain formation. An estimation shows
that such a process is possible from the
energy angle if the dislocation density
of the migrating boundary is greater than
about $10^{11}/cm^2$, a value which can readily be
reached.

Moreover, however, it is conceivable that
stress-induced grain boundary movement
occurs as a result of the marked increase
in the stresses effecting the grain boundaries
due to the dislocation blockage. On the
basis of the results obtained it is barely
possible to decide which process (strain
or stress-induced movement) is the decisive
one. Long-time anneals on samples with
serrated grain boundaries at 800 or 900°C
without stress produced no change in the
grain boundaries, so that it can be assumed
that the formation of the grain boundaries
is largely determined by the deformation
processes which occur during grain boundary
migration and less by a previously formed
dislocation structure.

Abstract

Studies were performed on the creep rupture
and creep behavior of binary V-Ti and ternary
V-Ti-Nb alloys in the temperature range above
650°C.

The results show the prominent influence
of the titanium on the creep rupture and
creep behavior of the alloys. For titanium
contents of above about 1 % the creep rupture ──→

strength drops for rising titanium concentrations, while the short-time high-temperature strength rises. This applies regardless of the niobium content. For increasing niobium contents the holding time is increased, but the influence of the niobium drops for rising holding times, so that the $\sigma_{B/10,000}$ values depend only slightly on the niobium content. The best vanadium alloys show long-time strength values which can barely be reached by any other materials. A marked dependence on the titanium content is also displayed by the creep activation energy in the temperature range around 650°C, while the niobium content has no influence upon this quantity.

The results of the studies indicate that the creep rupture and creep behavior of the alloys are also determined by the interactions between titanium and the interstitial impurities at high titanium contents.

References

1) H. Böhm und M. Schirra, J.Less-Common Metals, 12 (1967), 280

2) H. Böhm, W. Dienst, H. Hauck und H.J. Laue, ASTM Spec.Techn.
   Publ.

3) W. Burt et al., Argonne National Laboratory Reports
   ANL-6928 (1965) und ANL-7127 (1966)

4) "Alkali Metal Coolants", Symposium der IAEA, Wien 1967
   S. 45 und S. 63

5) H.U. Borgstedt, G. Drechsler und G. Frees, Werkstoffe und
   Korrosion 18 (1967) 894

6) Armour Research Foundation, WADCTR 52-145:
   W. Rostoker und M. Hansen, AD 13776, 1952;
   W. Rostoker, D.J. McPherson und M. Hansen, AD 56083, 1964;
   A.S. Yamamoto und W. Rostoker, AD 58609, 1955

7) W. Rostoker, A.S. Yamamoto und R.E. Riley, Trans.Am.Soc.
   Metals, 48 (1956) 560-78

8) K.F. Smith und R.S. Van Thyne, Argonne Nat.Lab.,
   ANL-5661, W-31-109-eng-38, 1957

9) R.J. Van Thyne, in W.R. Clough, Reactive Metals, Inter-
   science, New York/London, 1959, S. 403

10) B.R. Rajala und R.J. Van Thyne, Armour Research Foundation,
    Summary Rpt. 2165-6, AD 231512, NDas 59-6050-C, Dez. 1959

11) B.R. Rajala und R.J. Van Thyne, Armour Research Foundation,
    Final Rept. 2191-6, Dez. 1960

12) W. Pollack, E.C. Bishop, R.T. Begley und R.W. Buckman,
    Trans. ANS 10 (1967), 120

13) C.E. Lacy und C.J. Beck, Trans. ASM 48 (1956), 579

14) M. Rühle, persönl. Mitteilung

15) O.D. Sherby, Acta Met. 10 (1962), 135

16) C.R. Barrett, A.J. Ardell und O.D. Sherby, Trans. AIME 230 (1964), 200

17) A.W. Mullendore und N.J. Grant in "Structural Processes in Creep", Iron and Steel Institute, London, 1961, S. 44

18) H.C. Chang und N.J. Grant, Trans. AIME 194 (1952), 619

19) H. Brunner und N.J. Grant, Trans. AIME 218 (1960), 122

20) F.C. Monkman und N.J. Grant, Proc. ASTM 56 (1956), 593

21) O.D. Sherby, Acta Met. 10 (1962), 135

22) R.R. Vandervoort, Trans. AIME 242 (1968), 345

23) J. Weertman, J. Appl. Phys. 28 (1957), 1185

24) J. Weertman, Trans. AIME 218 (1960), 207

25) H. Böhm und F. Mir, J.Less-Common Metals 11 (1966), 408

26) Y.A. Rocher, L.A. Shepard und J.E. Dorn, Trans. AIME 215 (1959), 316

27) N.R. Borch, L.A. Shepard und J.E. Dorn, Trans. ASM 52 (1960), 494

28) H. Böhm und M. Schirra, Veröffentlichung demnächst

Table 1 : **List of alloys studied**

(in wt.%)

V - 3 Ti	V - 3 Ti - 10Nb	V - 1 Ti - 15Nb	
		V - 2 Ti - 15Nb	
		V - 3 Ti - 15Nb	V - 3 Ti - 20Nb
		V - 4 Ti - 15Nb	
V - 5 Ti	V - 5 Ti - 10Nb	V - 5 Ti - 15Nb	V - 5 Ti - 20Nb
	V - 7,5Ti - 10Nb	V - 7,5 Ti - 15Nb	V - 7,5 Ti - 20Nb
	V - 10 Ti - 10Nb	V - 10 Ti - 15Nb	V - 10 Ti - 20Nb
V - 20 Ti	V - 20 Ti - 10Nb		V - 20 Ti - 20Nb

Table 2 :   List of n- and k-values of alloys studied

Alloy	Test temperature	n	k
V - 3Ti	650°	10,3	$3,6 \cdot 10^{-21}$
V - 5Ti	650	9,0	$4,6 \cdot 10^{-17}$
V - 5Ti	700	4,7	$8,6 \cdot 10^{-11}$
V - 20Ti	650	4,2	$7,4 \cdot 10^{-12}$
V - 20Ti	700	2,9	$3,1 \cdot 10^{-7}$
V - 20Ti	800	1,9	$3,9 \cdot 10^{-5}$
V-3Ti-10Nb	650	6,4	$1,3 \cdot 10^{-14}$
V-5Ti-10Nb	650	5,5	$1,4 \cdot 10^{-12}$
V-7,5Ti-10Nb	650	4,4	$4,6 \cdot 10^{-10}$
V-10Ti-10Nb	650	3,4	$2,0 \cdot 10^{-8}$
V-20Ti-10Nb	650	3,1	$4,0 \cdot 10^{-7}$

Alloy	Test temperature	n	k
V-1Ti-15Nb	650°	11,3	$1,8 \cdot 10^{-23}$
V-2Ti-15Nb	650	7,4	$2,7 \cdot 10^{-16}$
V-3Ti-15Nb	650	6,6	$3,7 \cdot 10^{-15}$
V-4Ti-15Nb	650	5,6	$3,8 \cdot 10^{-13}$
V-5Ti-15Nb	650	5,3	$2 \cdot 10^{-12}$
V-7,5Ti-15Nb	650	4,5	$1,5 \cdot 10^{-10}$
V-10Ti-15Nb	650	3,9	$3,5 \cdot 10^{-9}$
V-3Ti-20Nb	650	8,1	$9,3 \cdot 10^{-18}$
V-5Ti-20Nb	650	6,9	$3,4 \cdot 10^{-15}$
V-7,5Ti-20Nb	650	4,7	$5,5 \cdot 10^{-11}$
V-10Ti-20Nb	650	6,2	$1,1 \cdot 10^{-13}$
V-20Ti-20Nb	650	4,6	$2,4 \cdot 10^{-10}$

V-3Ti
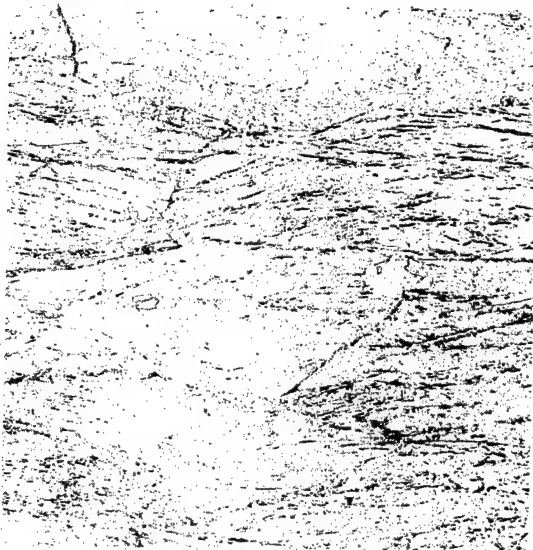
V-5Ti

HV$_{30}$ = 174-184
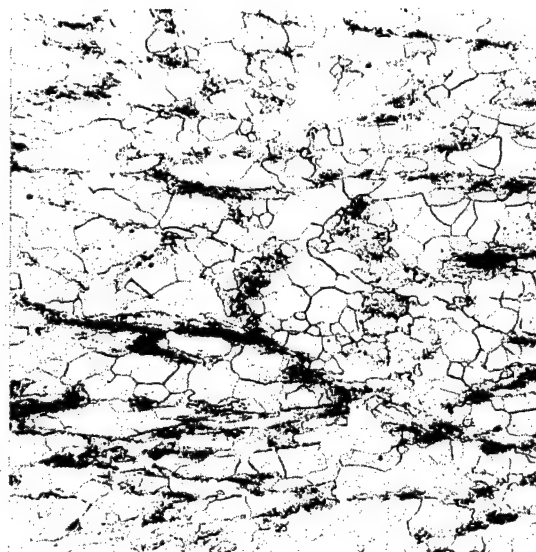
HV$_{30}$ = 136

V-1Ti-15Nb

HV$_{30}$ = 242-269

Ausgangsgefüge
===============

Zustand 900-950° 1^{h} Vakuum

Ätzung: Glyzerin: HF : HNO$_3$

3   : 2 : 1

These photographs reduced 10% in printing process.

V = x 100

V-20Ti

V-3Ti-10Nb   Bild 1b

HV_{30} = 197-202

HV_{30} = 244

These photographs reduced 10% in printing process.

V-2Ti-15Nb

V-3Ti-15Nb

HV_{30} = 242-250
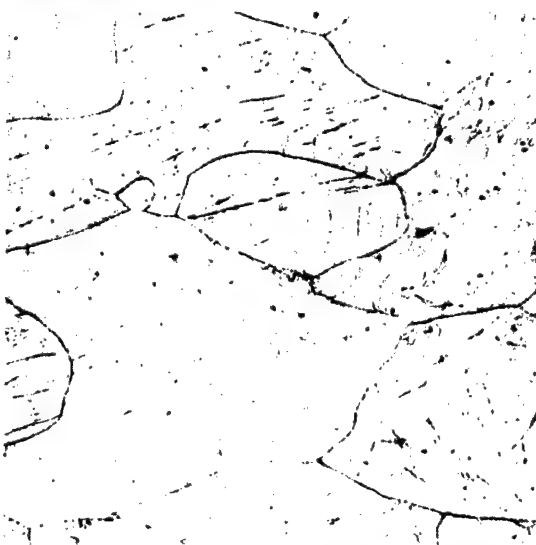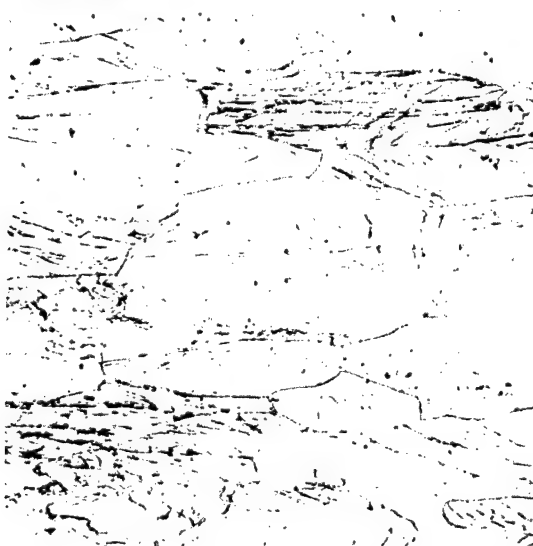
HV_{30} = 288

V-3Ti-20Nb

HV_{30} = 288   x 100

V-5Ti-10Nb

V-7,5Ti-10Nb

HV_{30} = 244

HV_{30} = 230-250

V-4Ti-15Nb

V-5Ti-15Nb

HV_{30} = 239-242
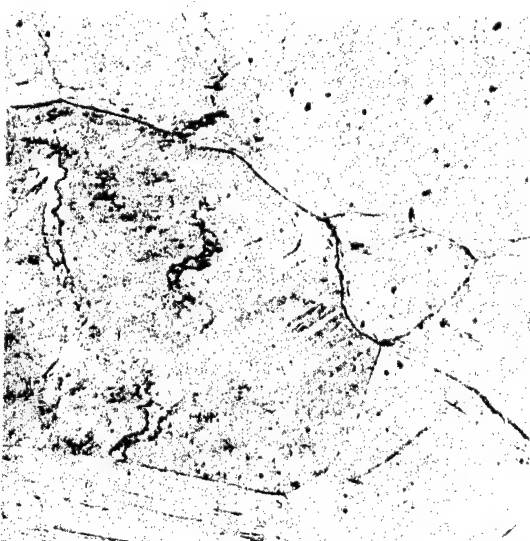
HV_{30} = 249-260

V-5Ti-20Nb

V-7,5Ti-20Nb

HV_{30} = 230-237

HV_{30} = 271-277 x 100

These photographs reduced 10% in printing process.

V-10Ti-10Nb

V-20Ti-10Nb

HV_{30} = 216-226
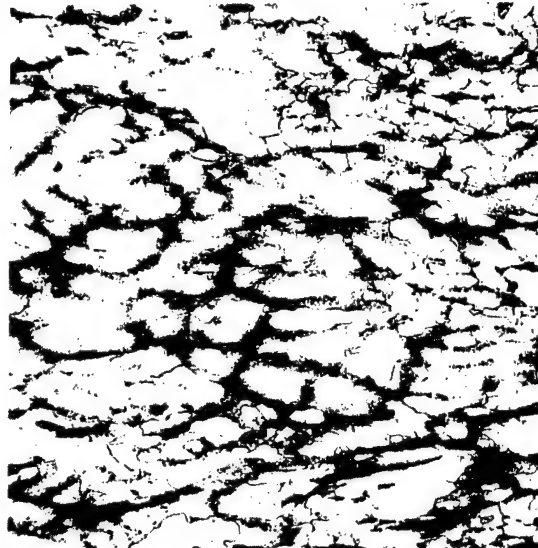
HV_{30} = 230-250

V-7,5Ti-15Nb

V-10Ti-15Nb

HV_{30} = 256

HV_{30} = 252-257

These photographs reduced 10% in printing process.

V-10Ti-20Nb

V-20Ti-20Nb

HV_{30} = 275-283

HV_{30} = 257-260 x 100

3 Zonen-Heizofen

3 fach Thermoele-ment

M8

53

25

5,0

3
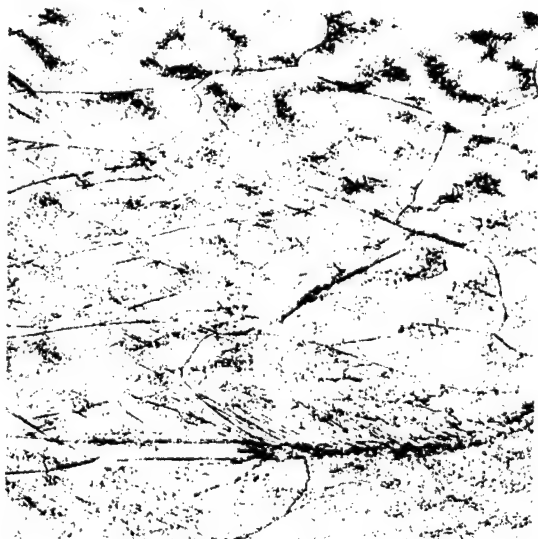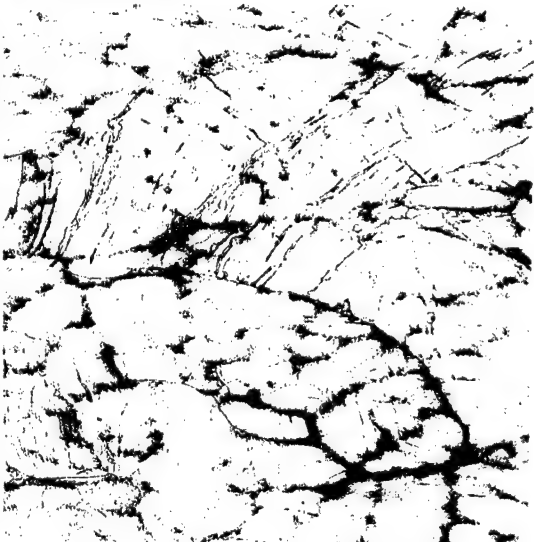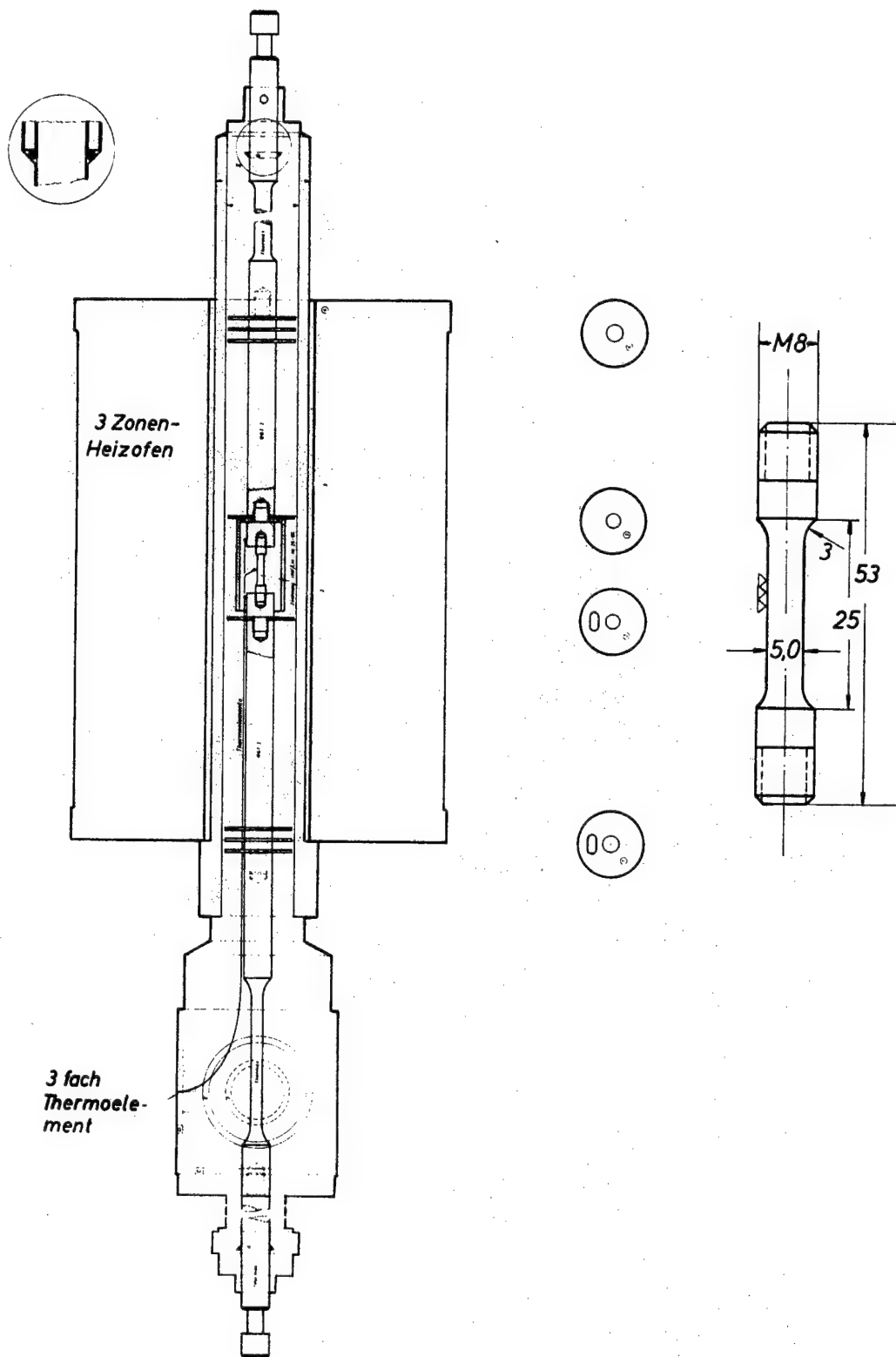
Bild 2 :   *Probenform und Teststrecke für Vakuum-Zeitstandversuche*
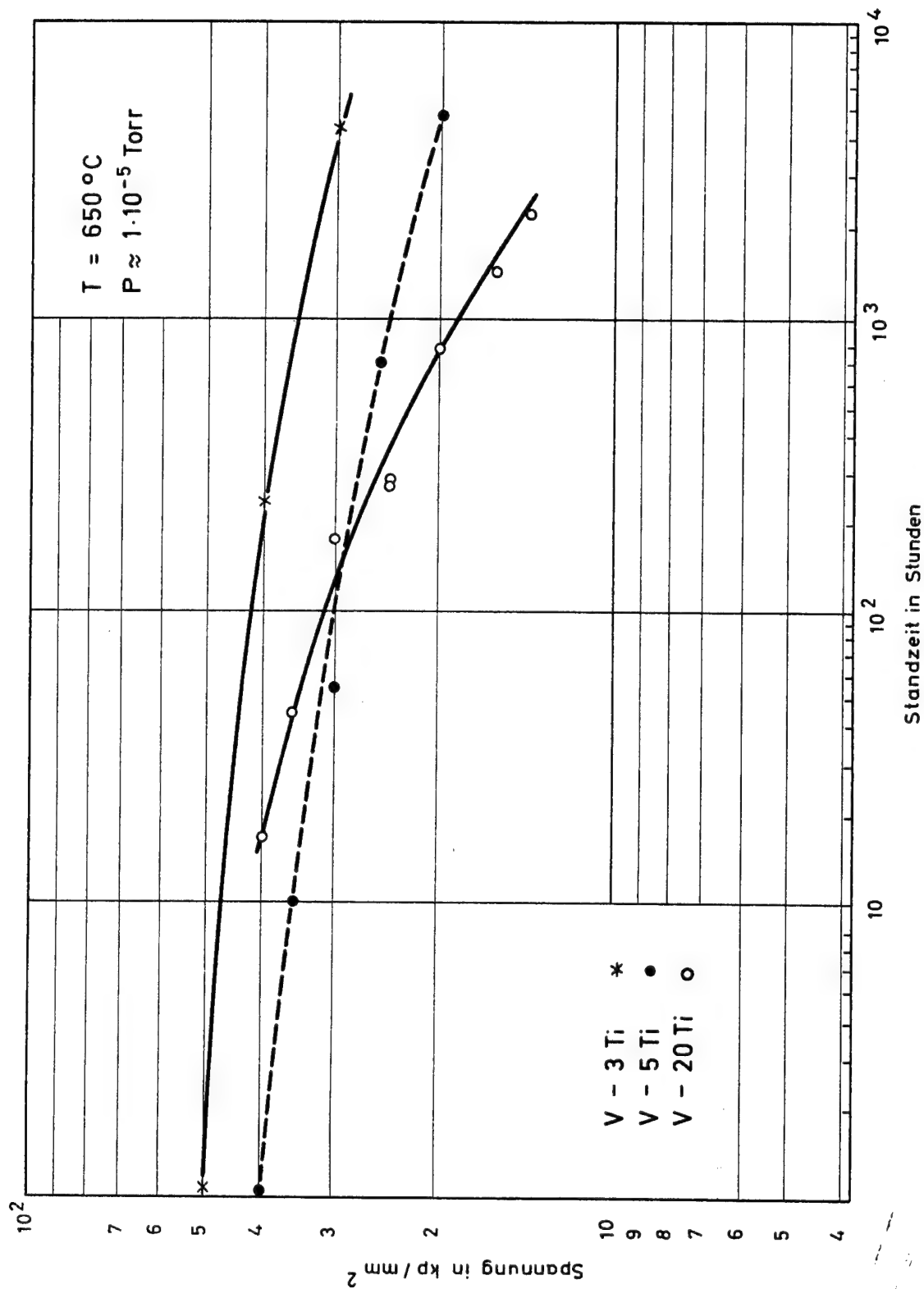
Bild 3: Einfluß des Ti-Gehaltes auf die Zeitstandfestigkeit von V-Legierungen
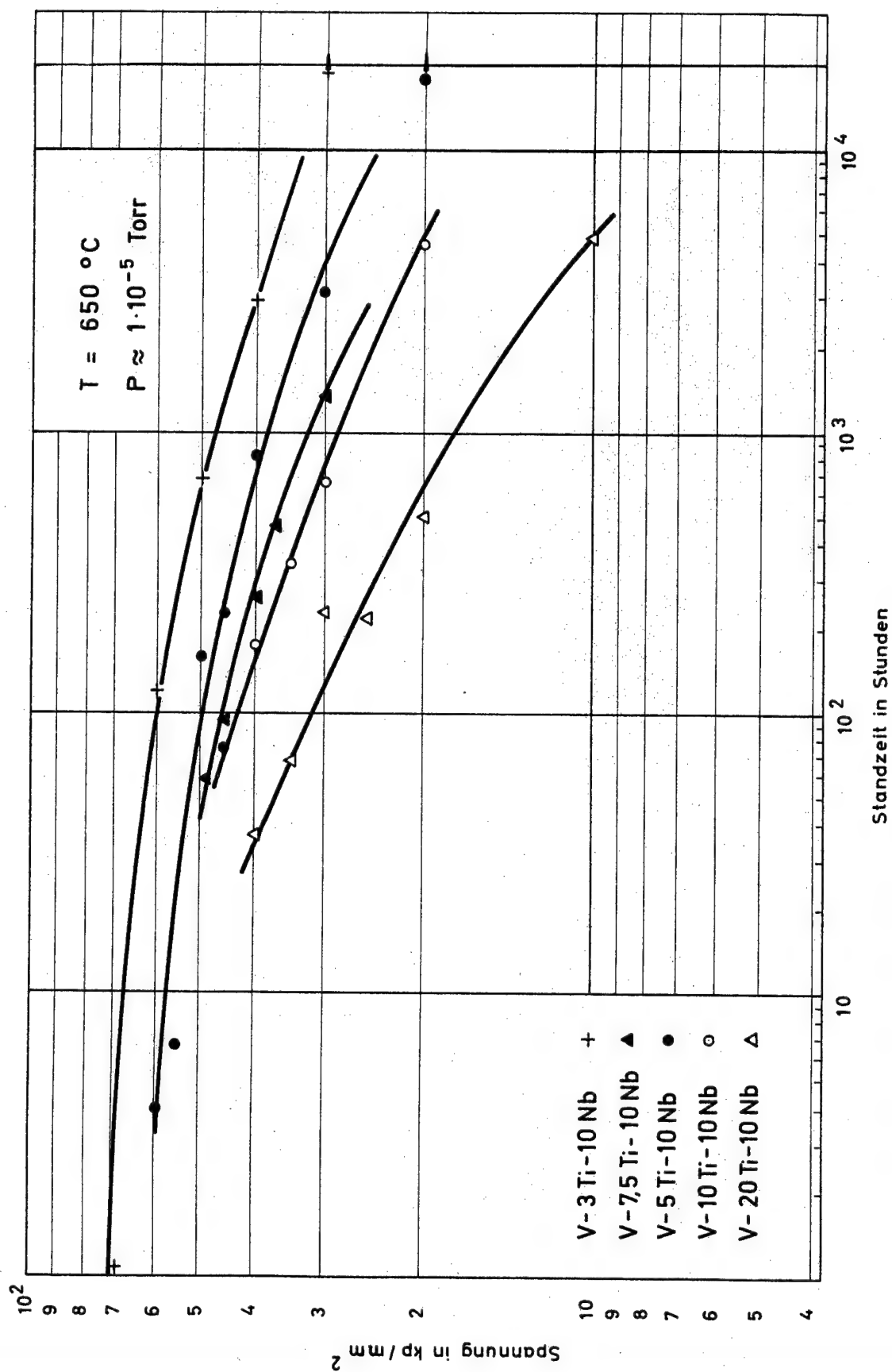
Bild 4 : Einfluß des Ti-Gehaltes auf die Zeitstandfestigkeit von V-10Nb - Legierungen

Legend:

Symbol	Legierung
+	V - 3 Ti - 10 Nb
▲	V - 7,5 Ti - 10 Nb
●	V - 5 Ti - 10 Nb
○	V - 10 Ti - 10 Nb
△	V - 20 Ti - 10 Nb

$T = 650\ °C$
$P \approx 1 \cdot 10^{-5}$ Torr

x-axis: Standzeit in Stunden

y-axis: Spannung in kp/mm^2
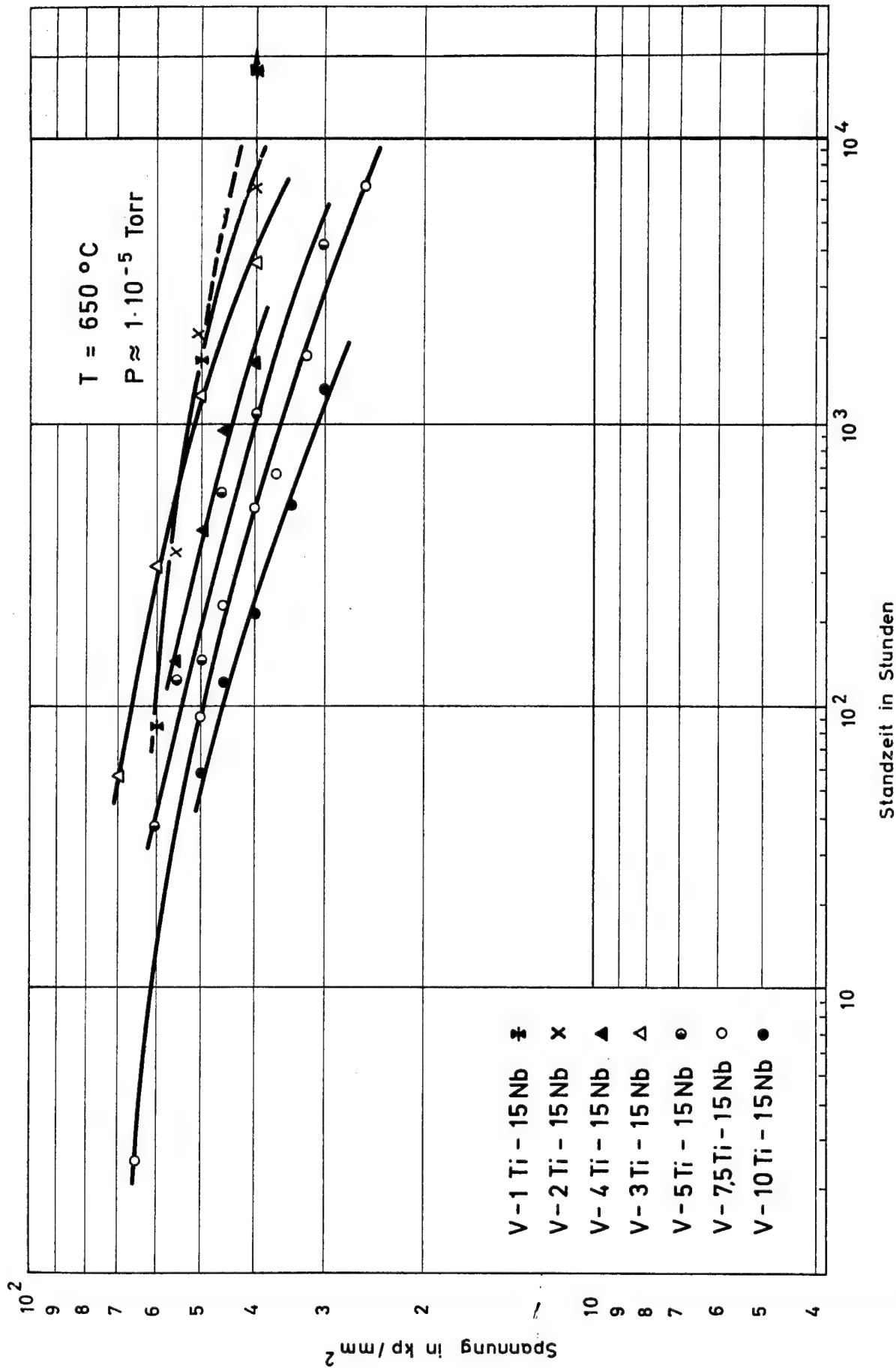
Bild 5 : Einfluß des Ti-Gehaltes auf die Zeitstandfestigkeit von V-15 Nb - Legierungen
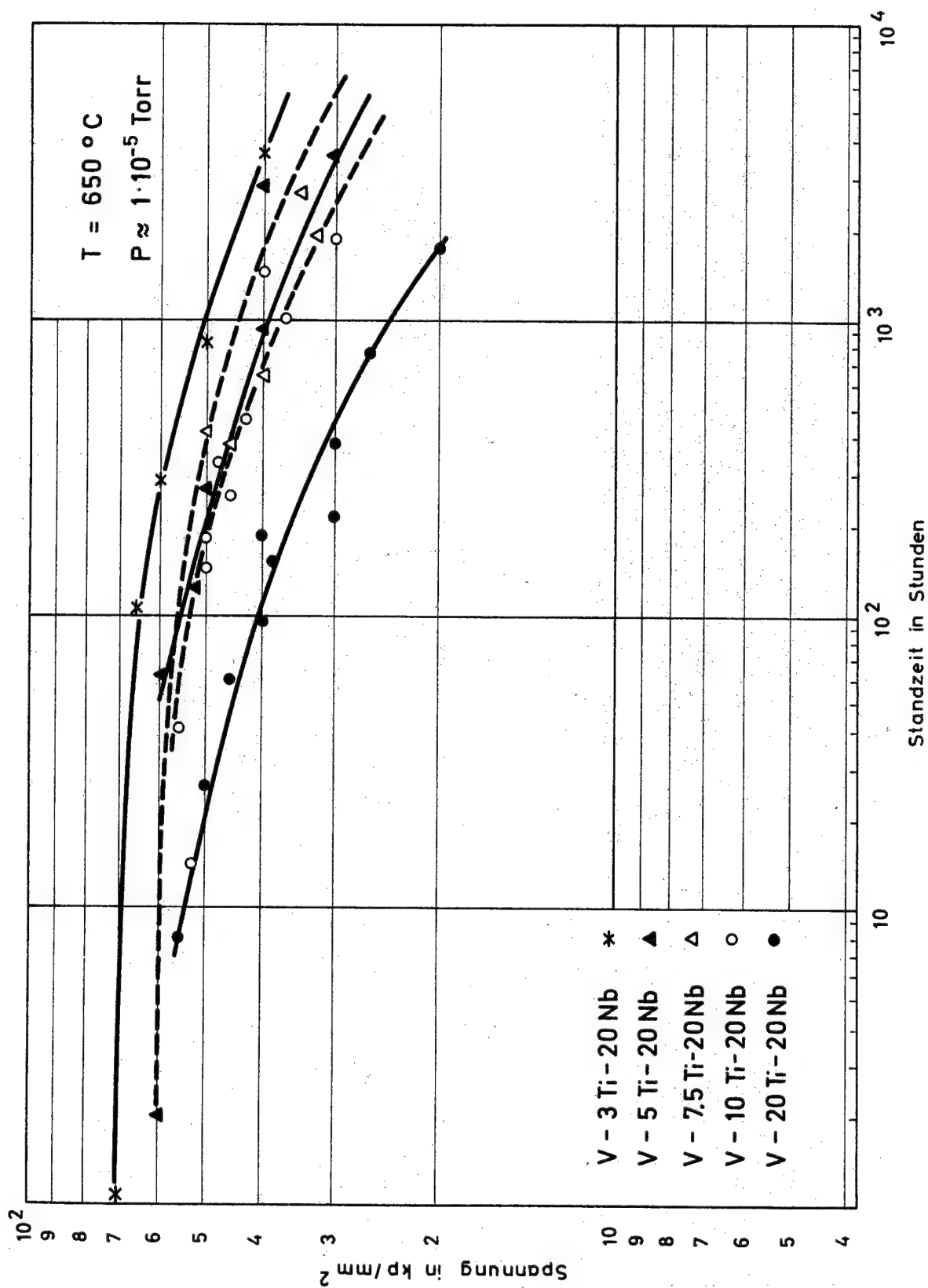
Bild 6: Einfluß des Ti-Gehaltes auf die Zeitstanfestigkeit von V-20Nb-Legierungen

Legend (within figure):

T = 650 °C
P ≈ 1·10⁻⁵ Torr

*   V – 3 Ti – 20 Nb
▲   V – 5 Ti – 20 Nb
△   V – 7.5 Ti – 20 Nb
○   V – 10 Ti – 20 Nb
●   V – 20 Ti – 20 Nb

Axis labels:
Spannung in kp/mm²
Standzeit in Stunden

$G_{B/100}$
in kp/mm²

% Ti

% Nb
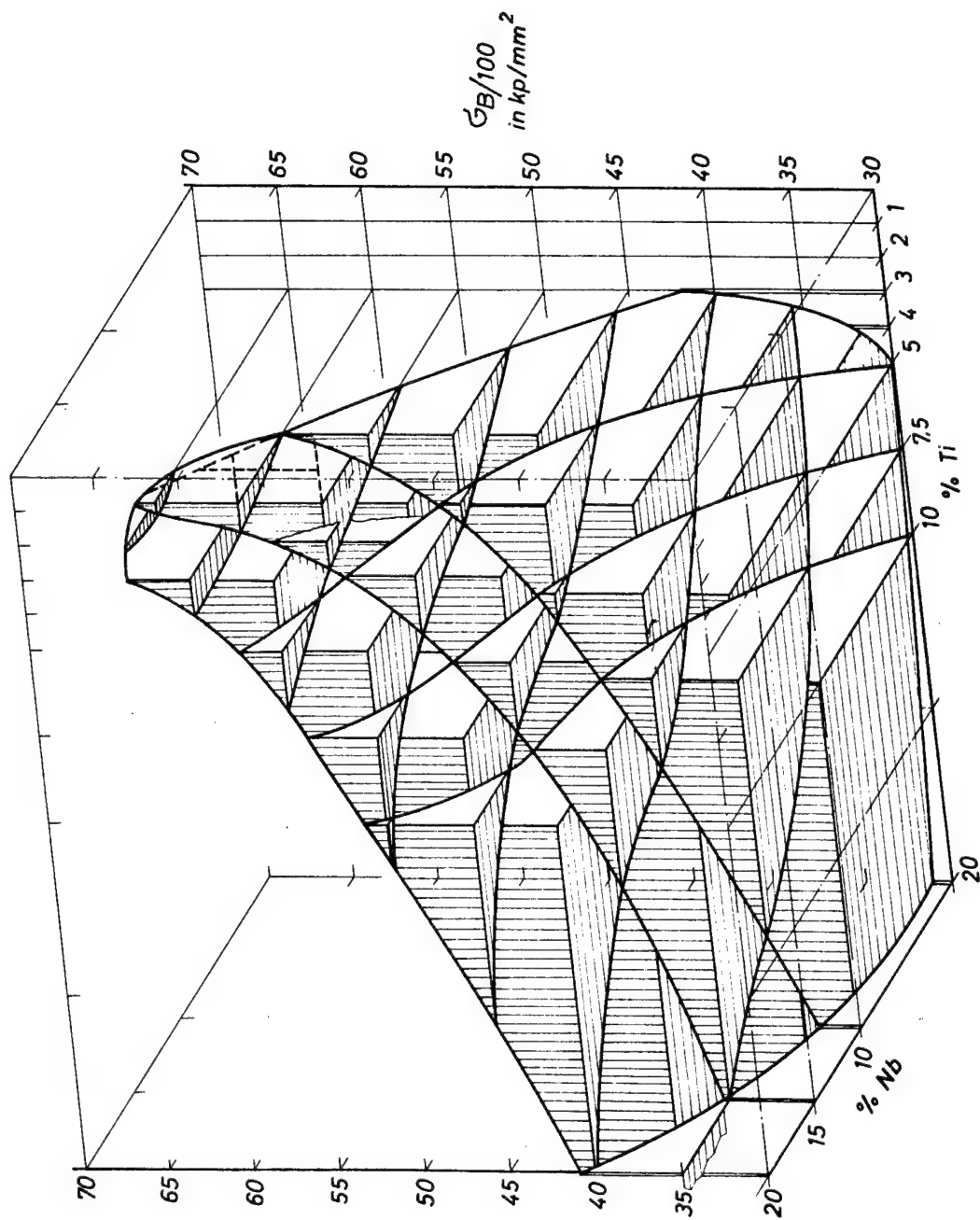
Bild 7 : 100-Stunden-Zeitstandfestigkeit von Vanadin-Basis-Legierungen in Abhängigkeit vom

Titan- und Niob-Gehalt   T = 650°C
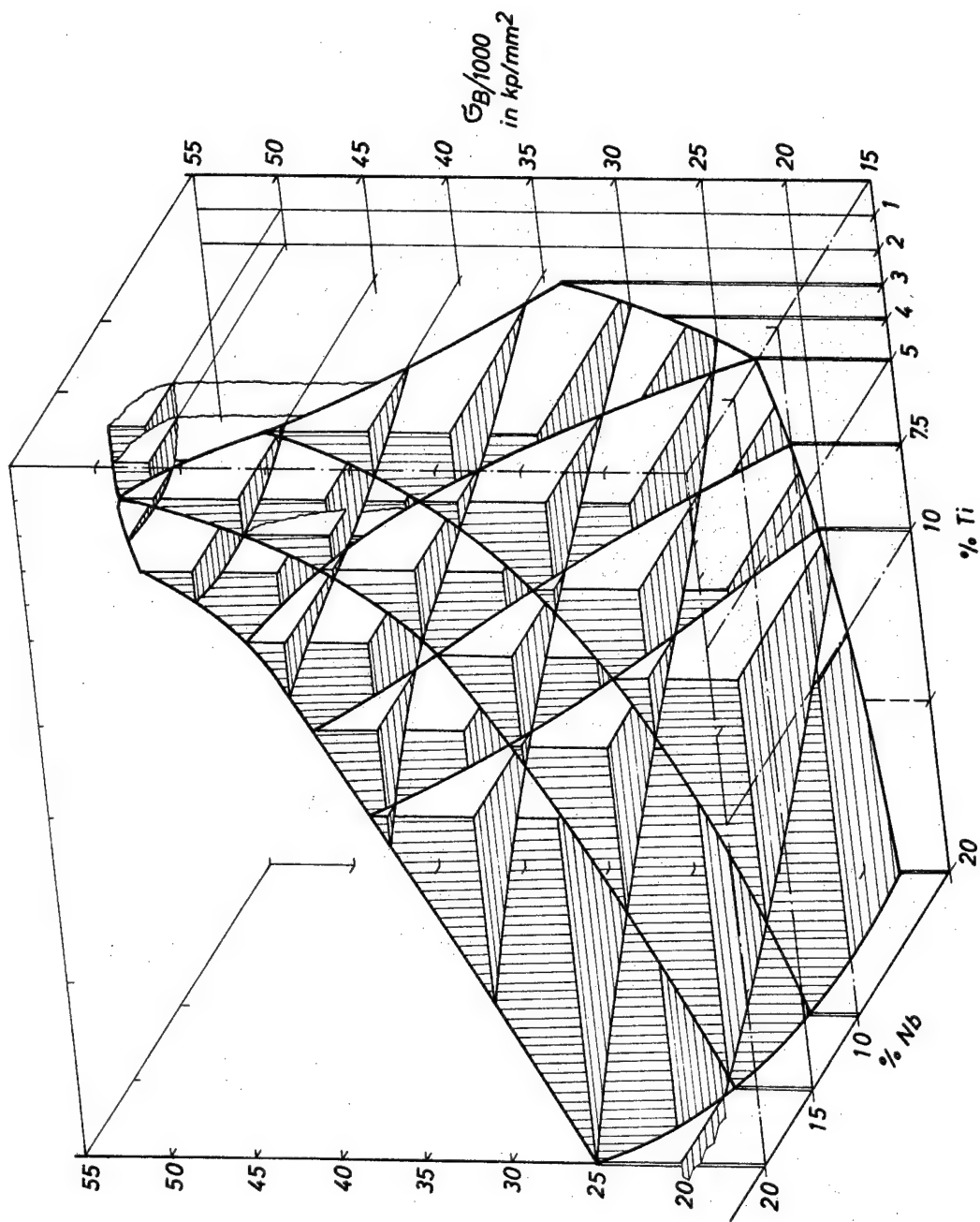
Bild 8 : 1000-Stunden-Zeitstandfestigkeit von Vanadin-Basis-Legierungen in Abhängigkeit vom Titan- und Niob-Gehalt (T = 650° C)
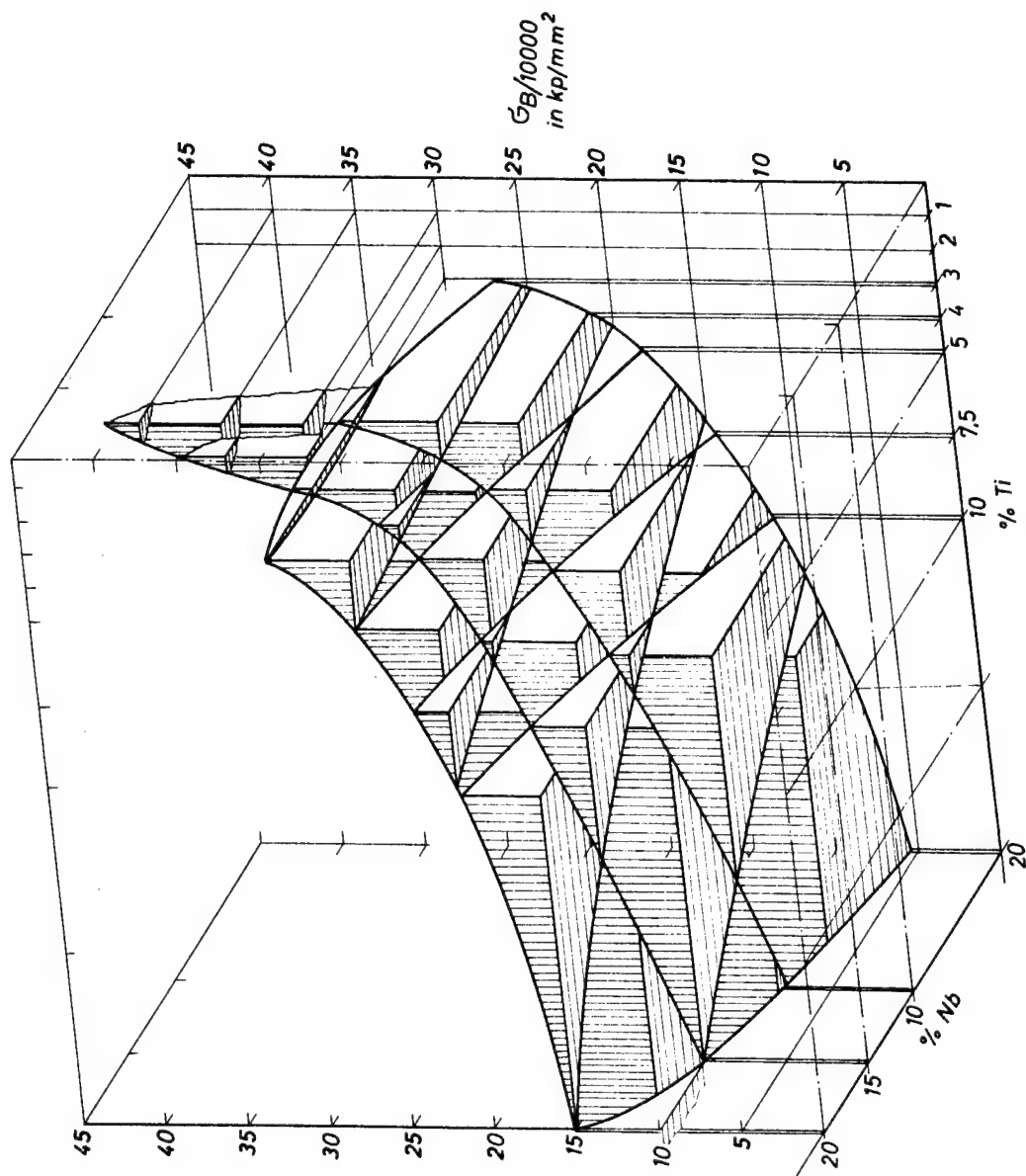
Bild 9 : 10000-Stunden-Zeitstandfestigkeit von Vanadin-Basis-Legierungen in Abhängigkeit vom Titan- und Niob-Gehalt (T= 650°C)
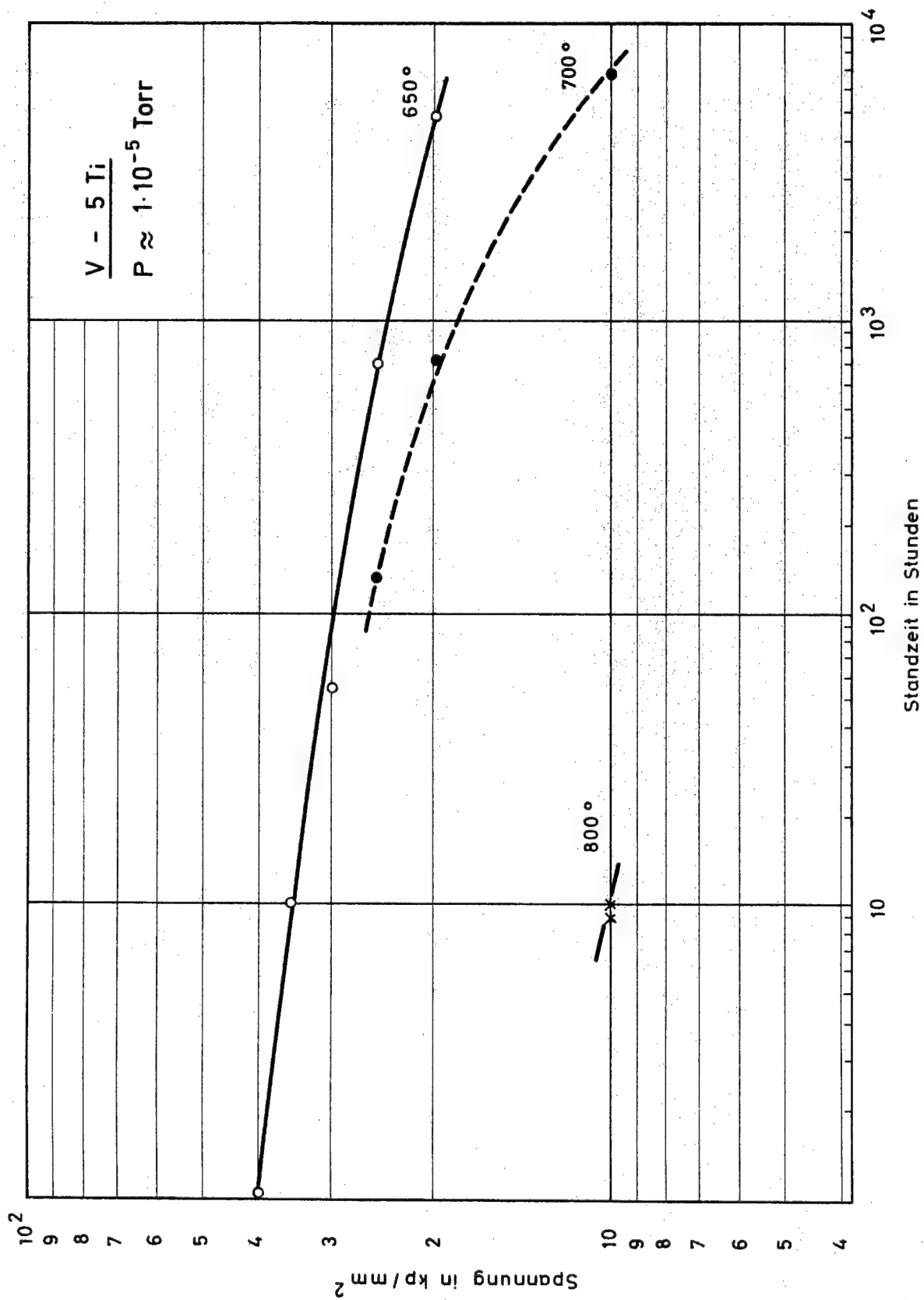
Bild *10* :   Einfluß der Prüftemperatur auf die Zeitstandfestigkeit von V–5 Ti
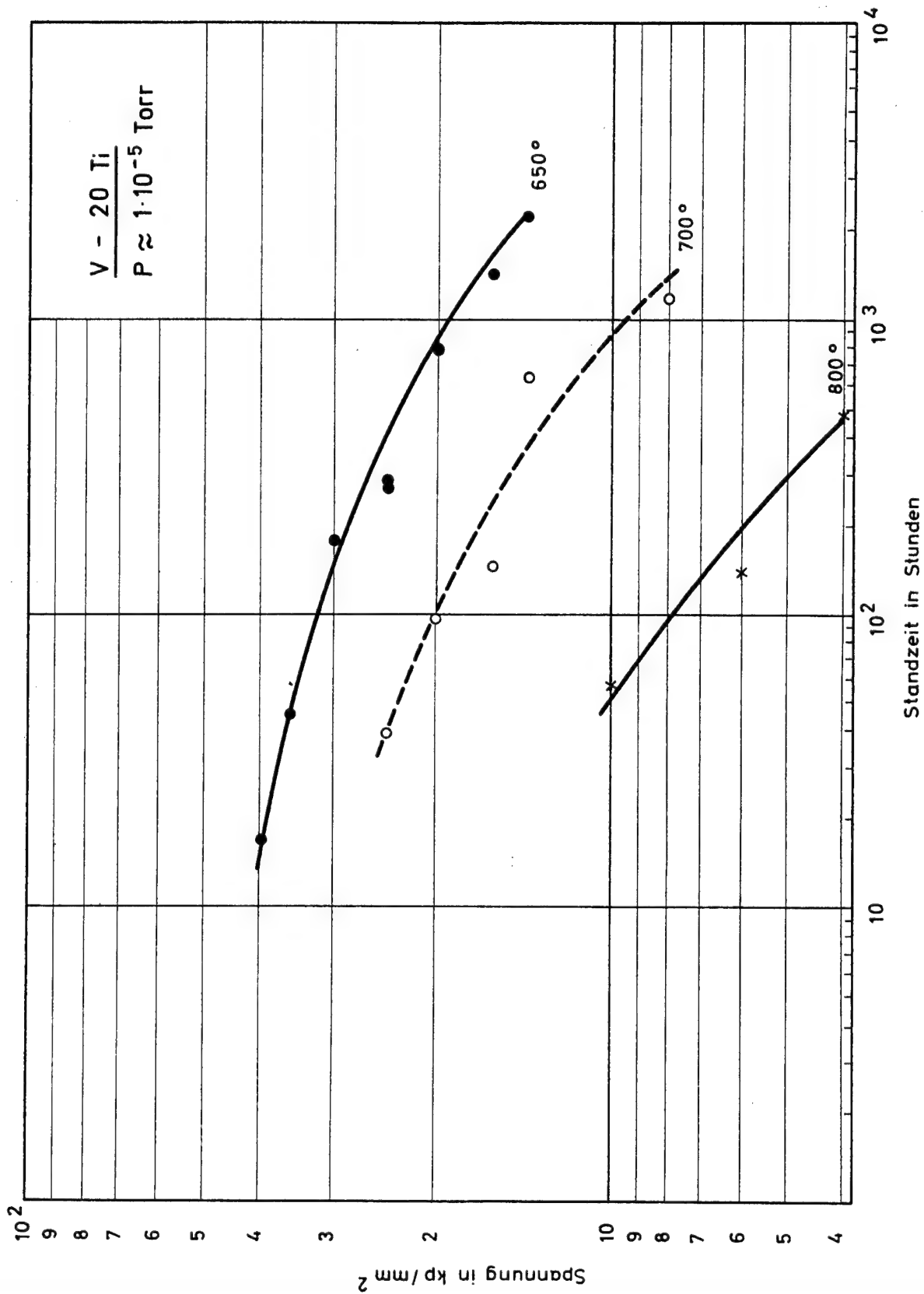
Bild *11*: Einfluß der Prüftemperatur auf die Zeitstandfestigkeit von V – 20 Ti
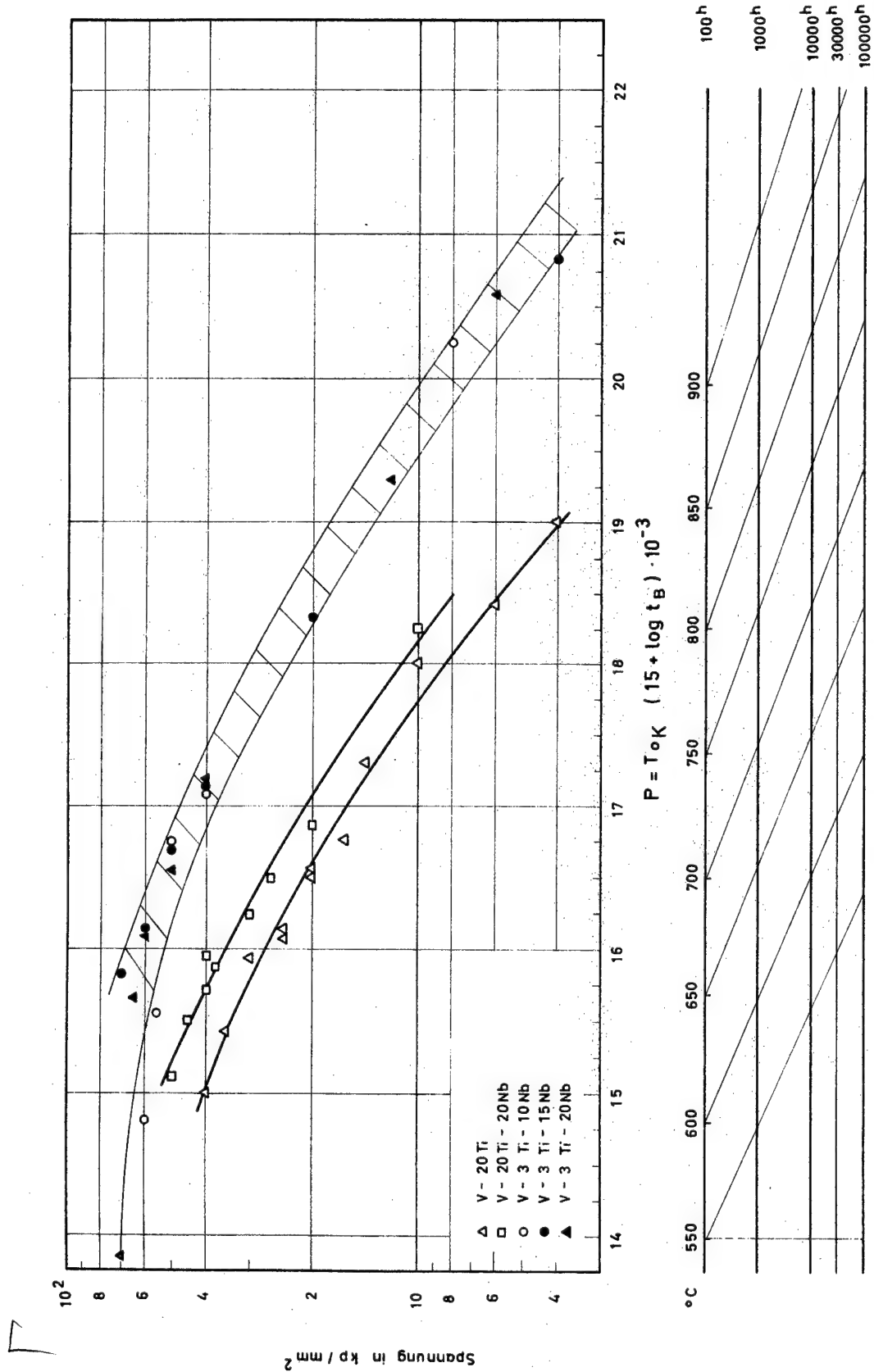
Bild 12 : Zeitstandfestigkeit in Abhängigkeit vom Larson - Miller - Parameter
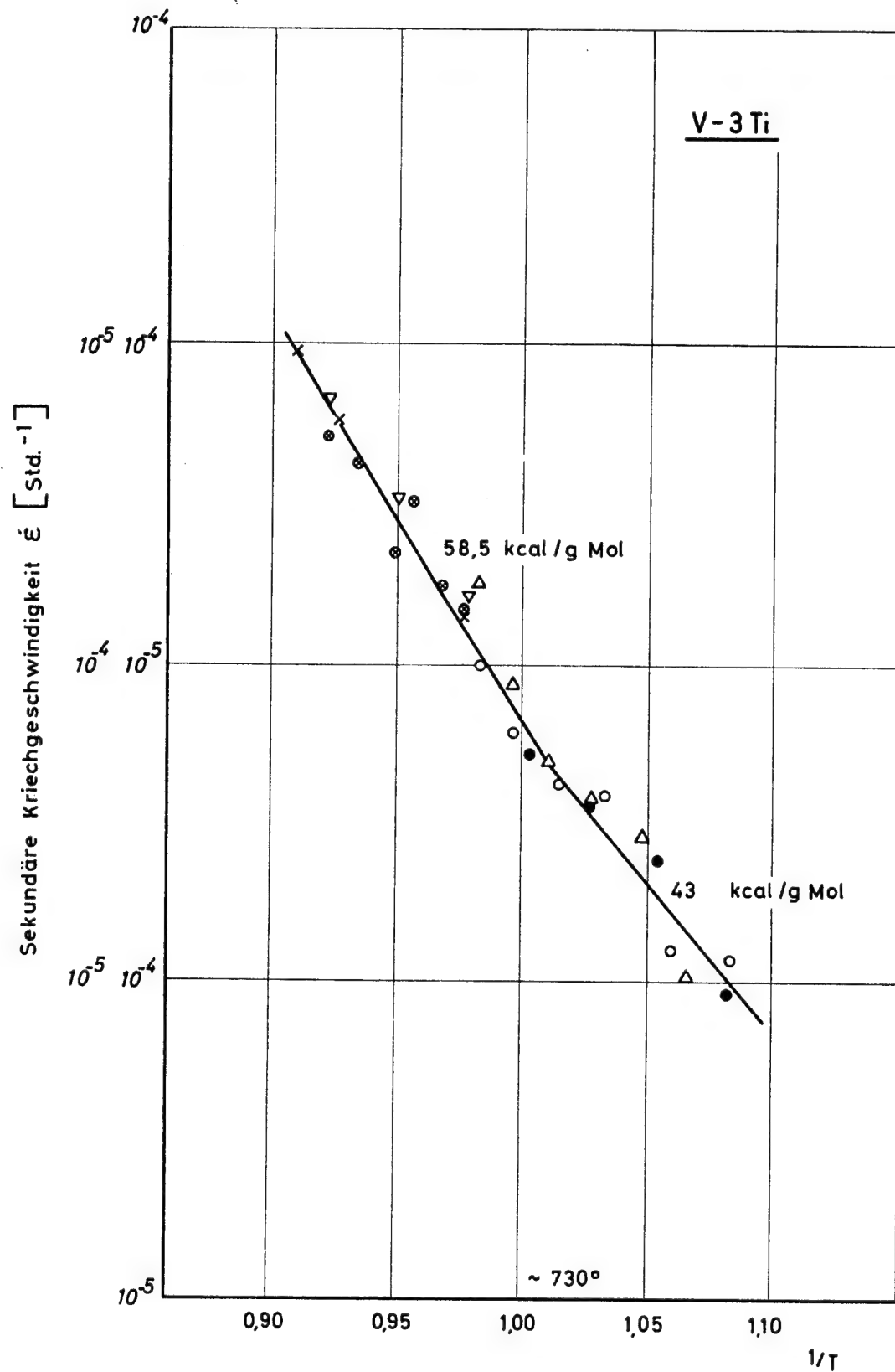
Bild *13* :   Kriechgeschwindigkeit in Abhängigkeit von der Temperatur
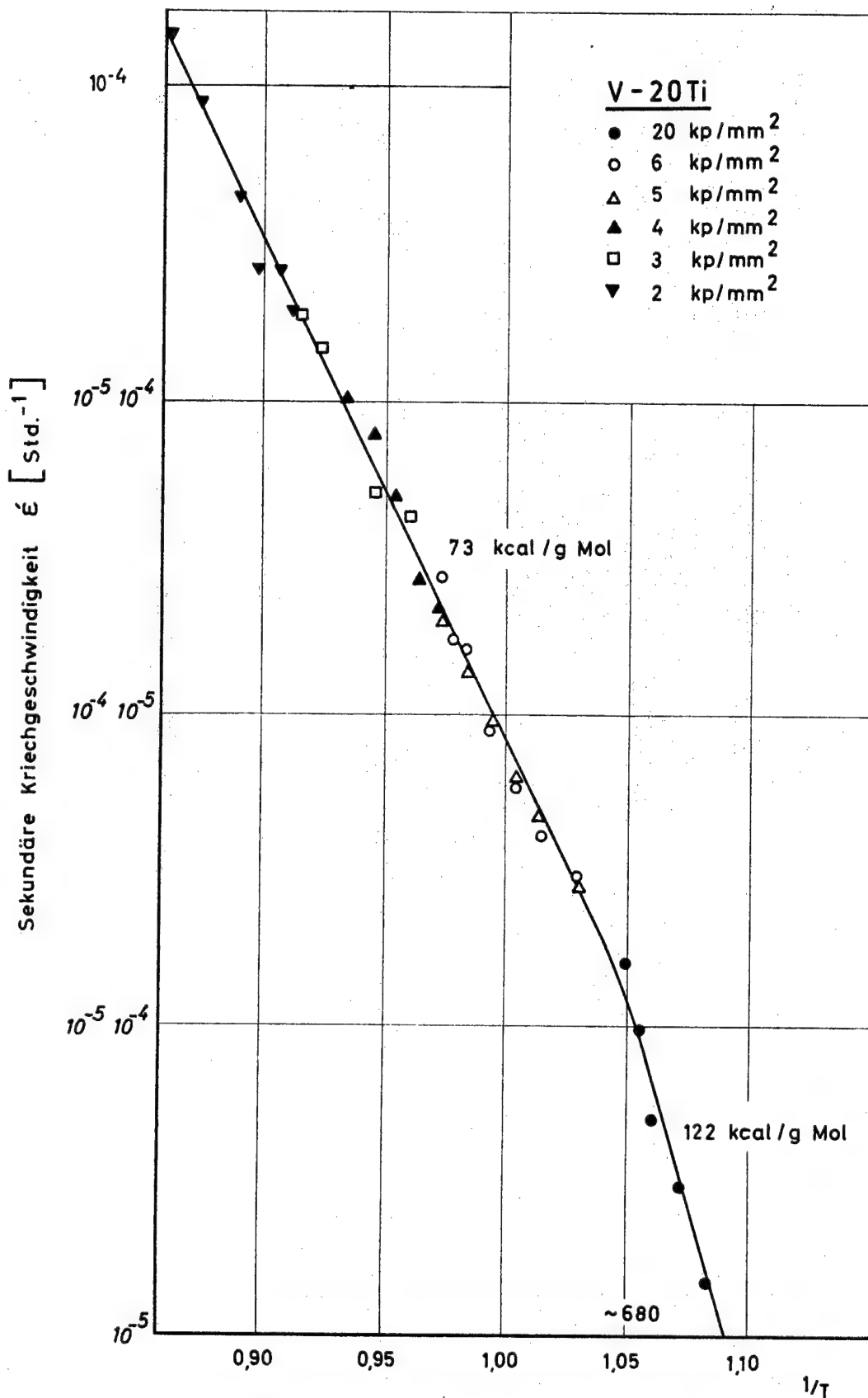
(Vakuum≈1·10^{-5} Torr)

Bild 14 : Kriechgeschwindigkeit in Abhängigkeit von der Temperatur
(Vakuum ≈ 1·10⁻⁵ Torr)
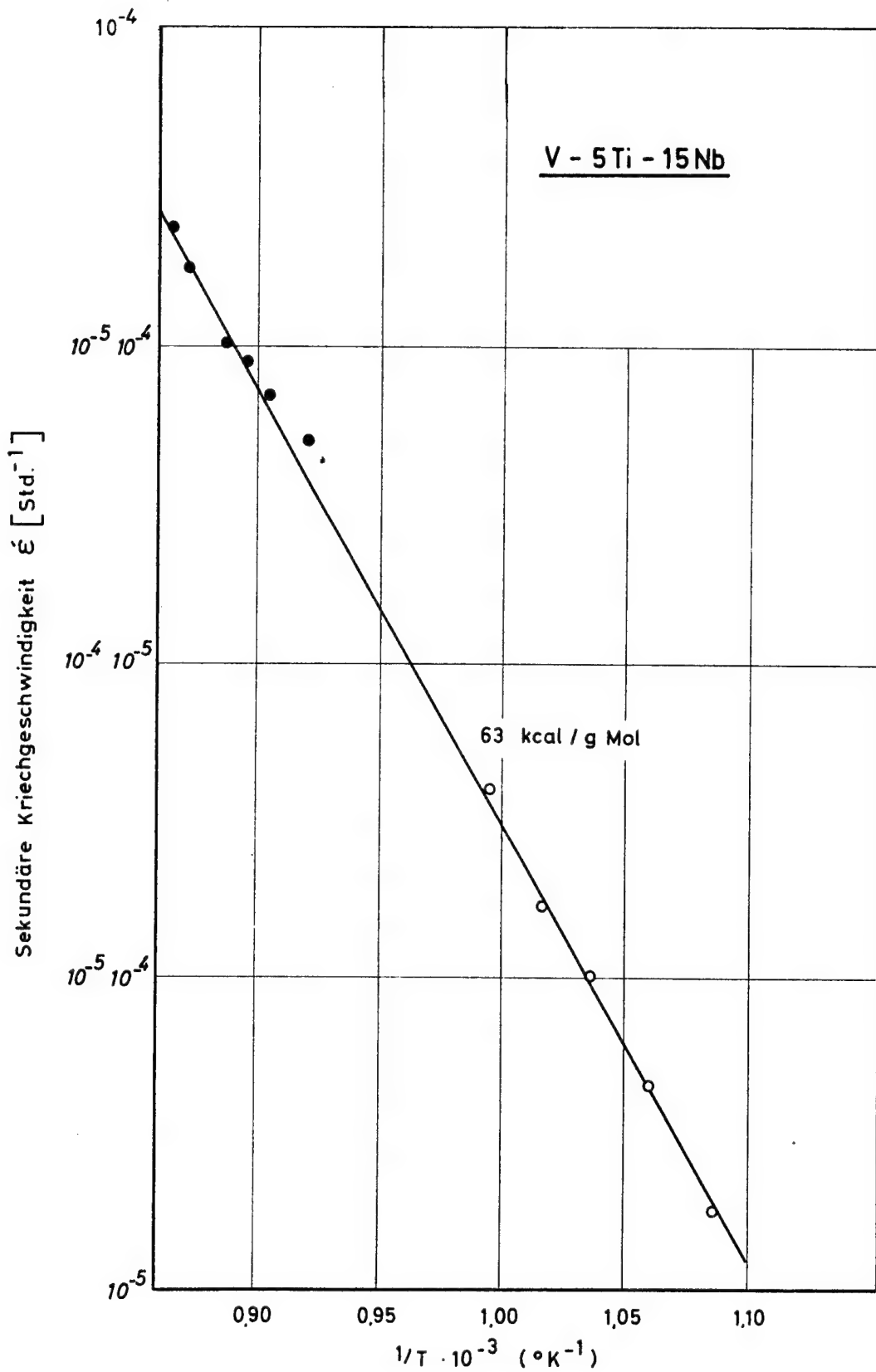
Bild *15* : Kriechgeschwindigkeit in Abhängigkeit von der Temperatur
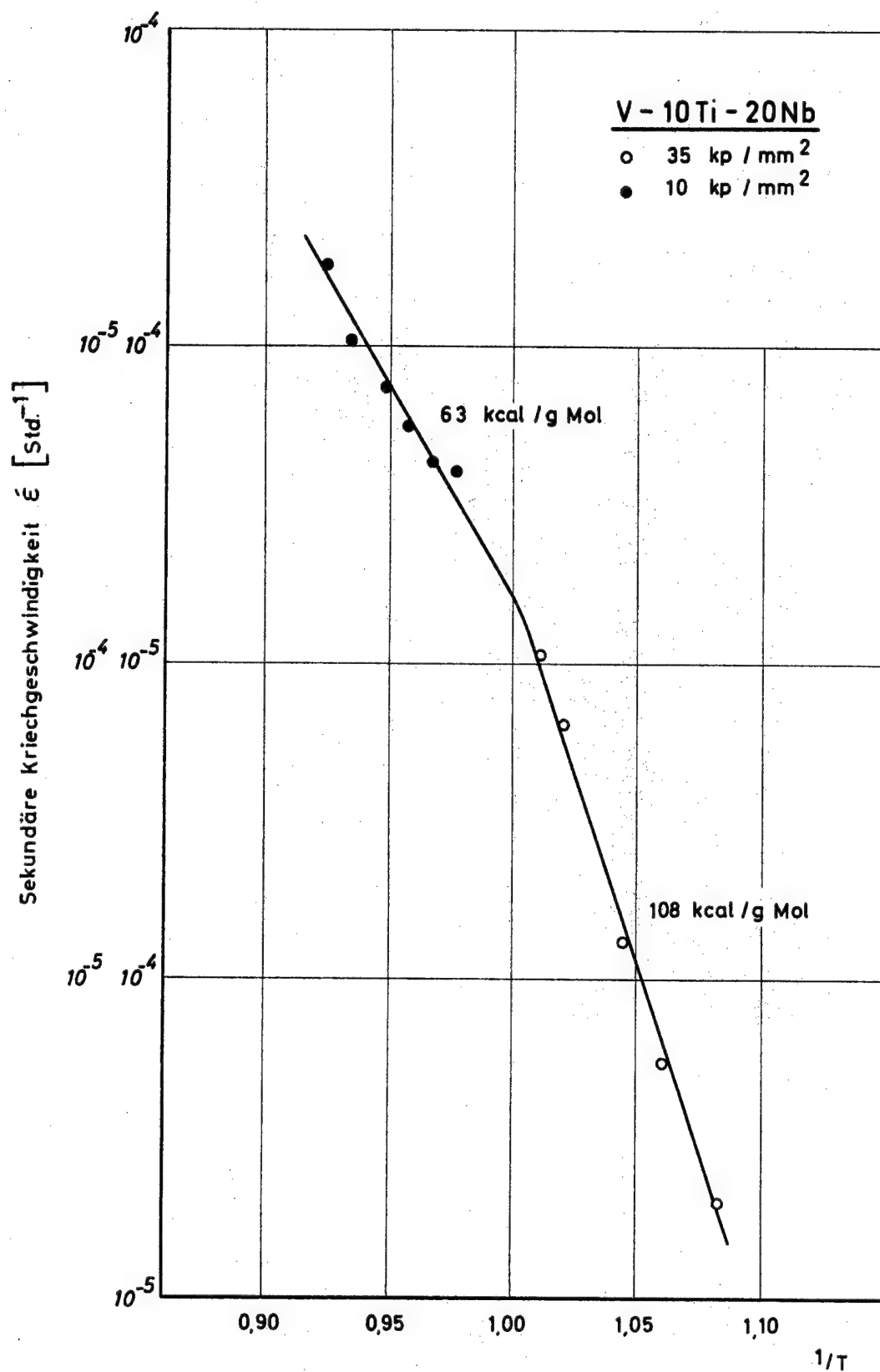
(Vakuum ≈ 1·10⁻⁵ Torr)

Bild 16: Kriechgeschwindigkeit in Abhängigkeit von der Temperatur
(Vakuum ≈ 1·10^{-5} Torr)
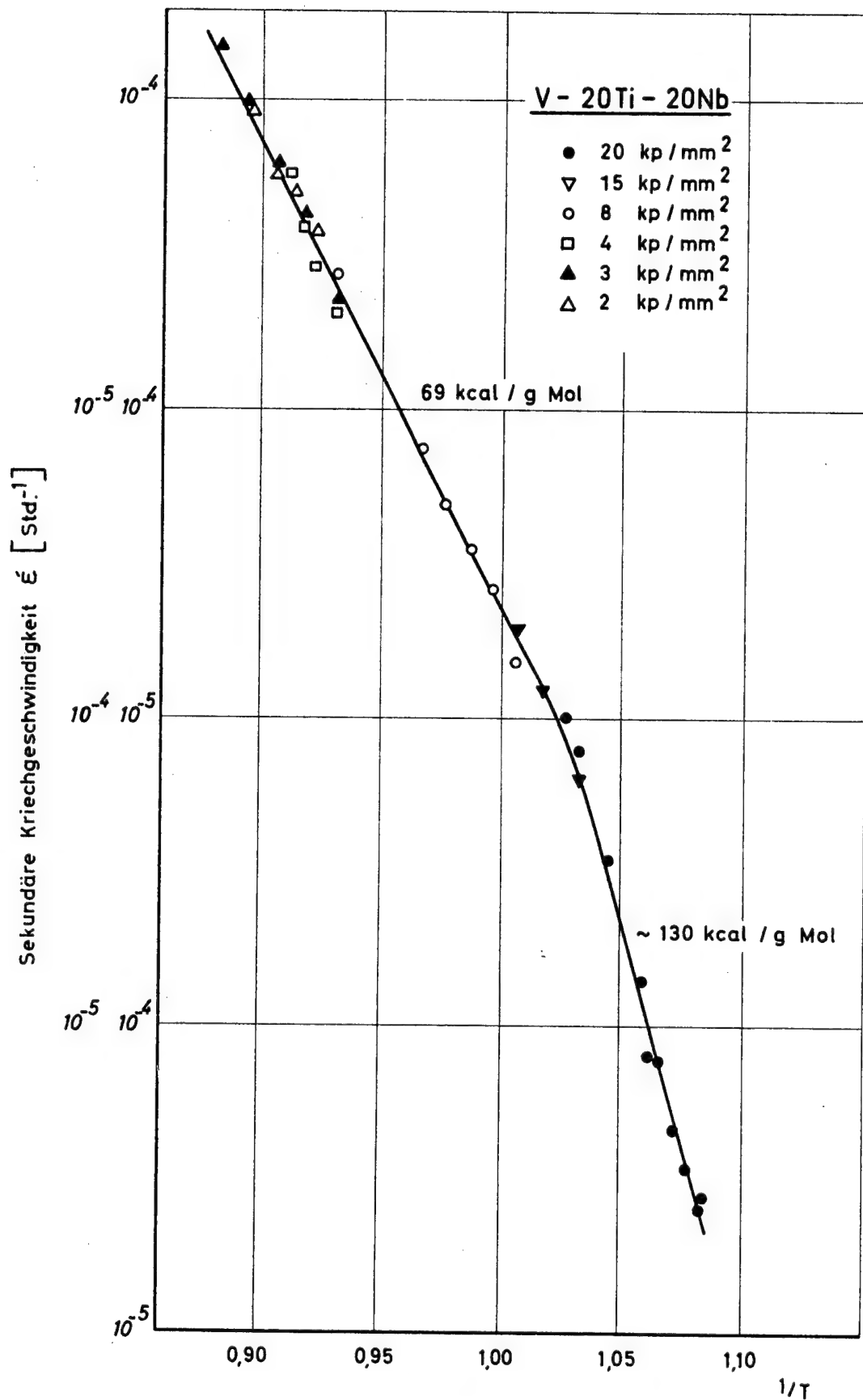
Bild *17* : Kriechgeschwindigkeit in Abhängigkeit von der Temperatur
(Vakuum ≈ 1 · 10⁻⁵ Torr)
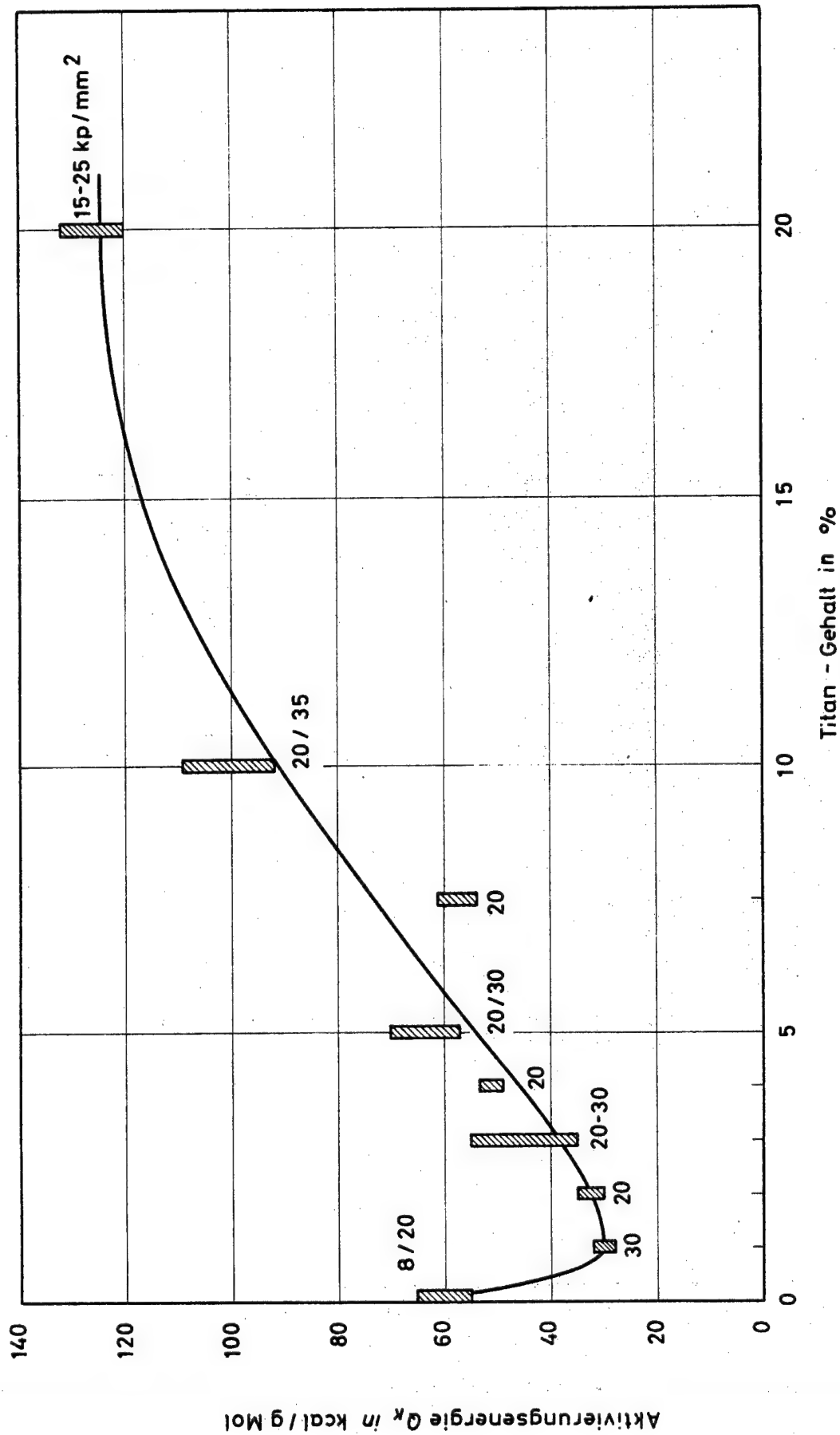
Bild 18 : Einfluß des Titan - Gehaltes auf die Aktivierungsenergie des Kriechens binärer und ternärer V - Legierungen (Temp.- Bereich 650 - 700 °C)
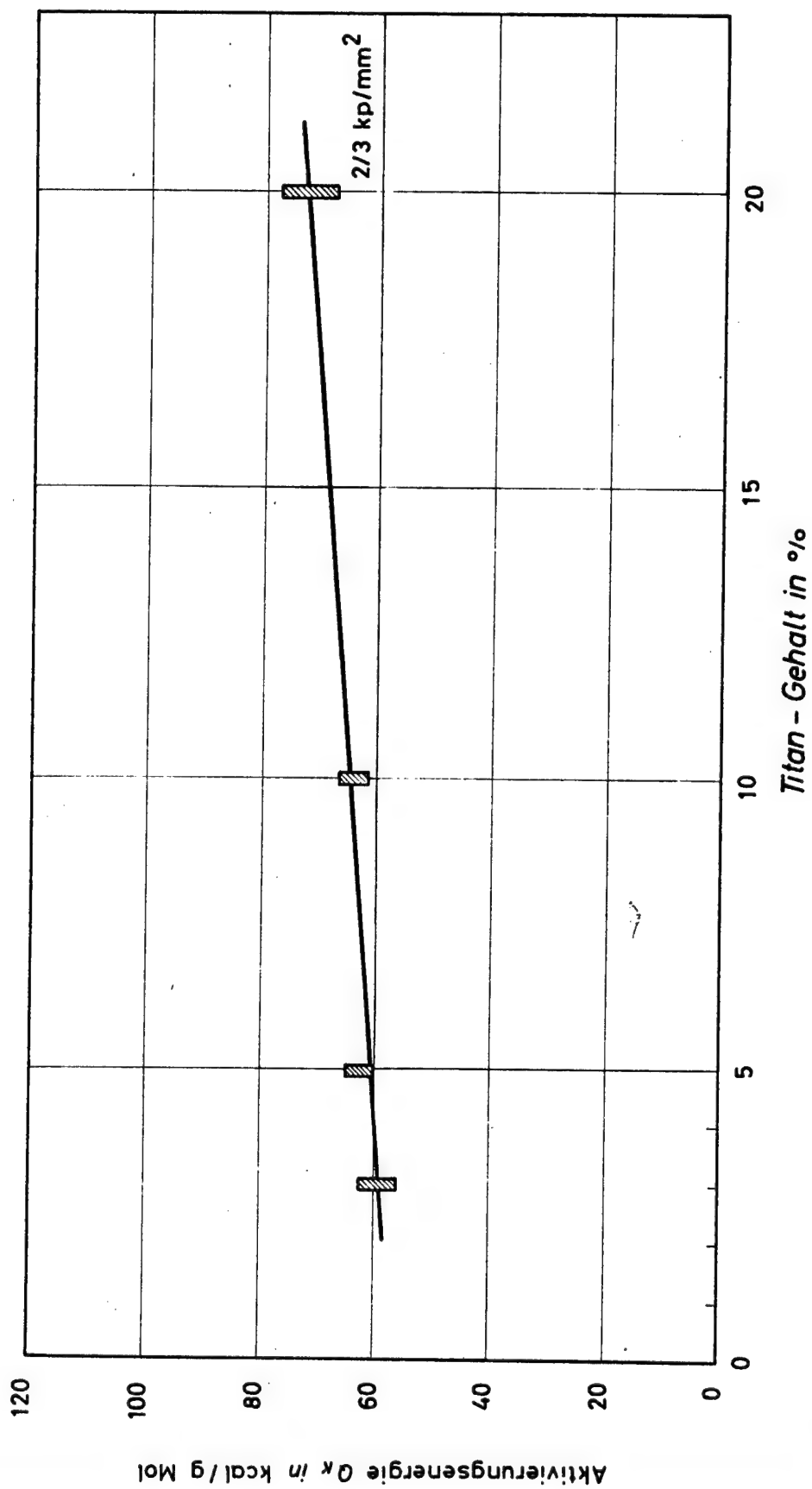
Bild 19: Einfluß des Titan – Gehaltes auf die Aktivierungsenergie des Kriechens bei Temperaturen > 750°
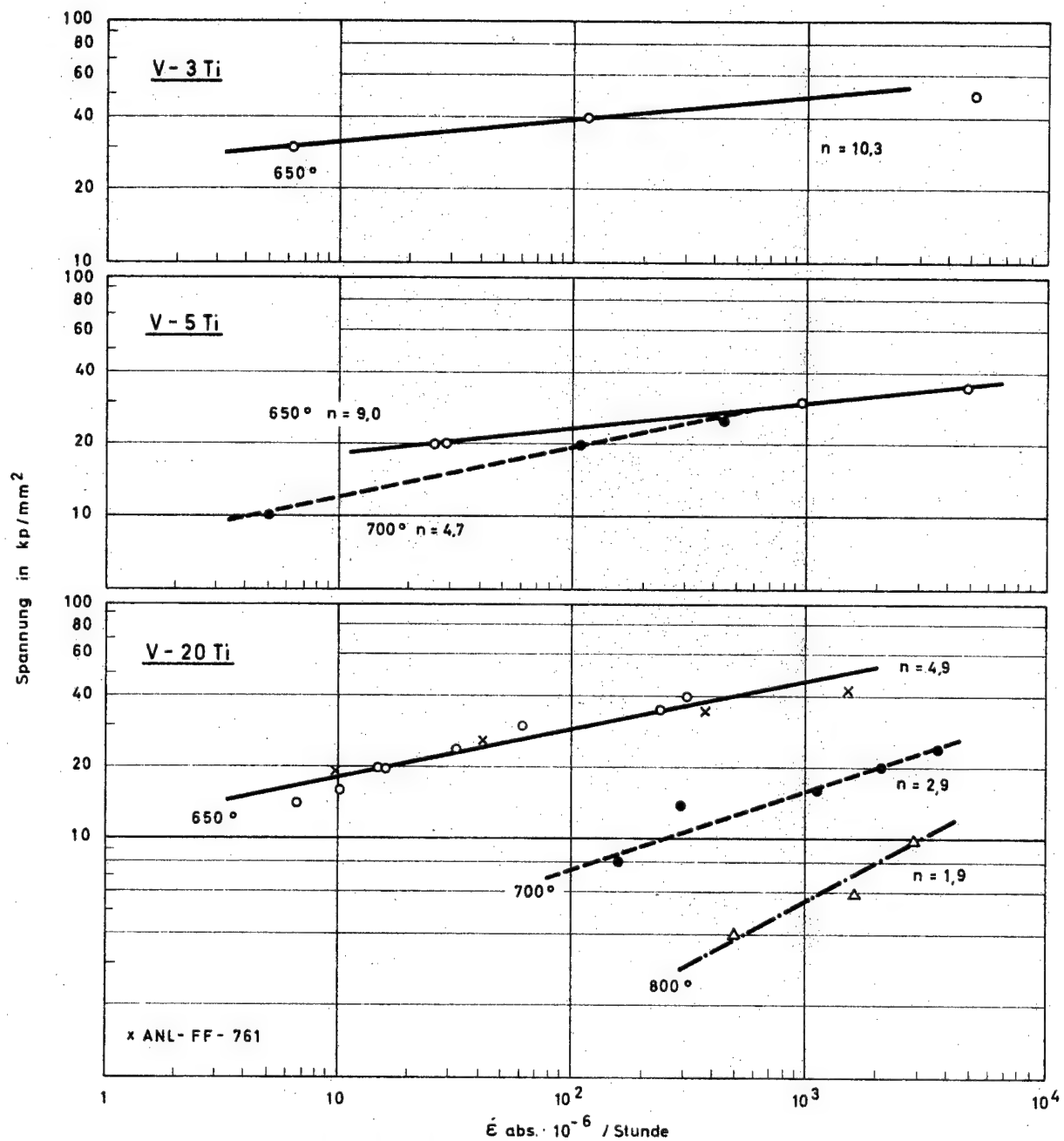
Bild 20: Abhängigkeit der sekundären Kriechgeschwindigkeit von der Spannung
(P ≈ 1 · 10⁻⁵ Torr)
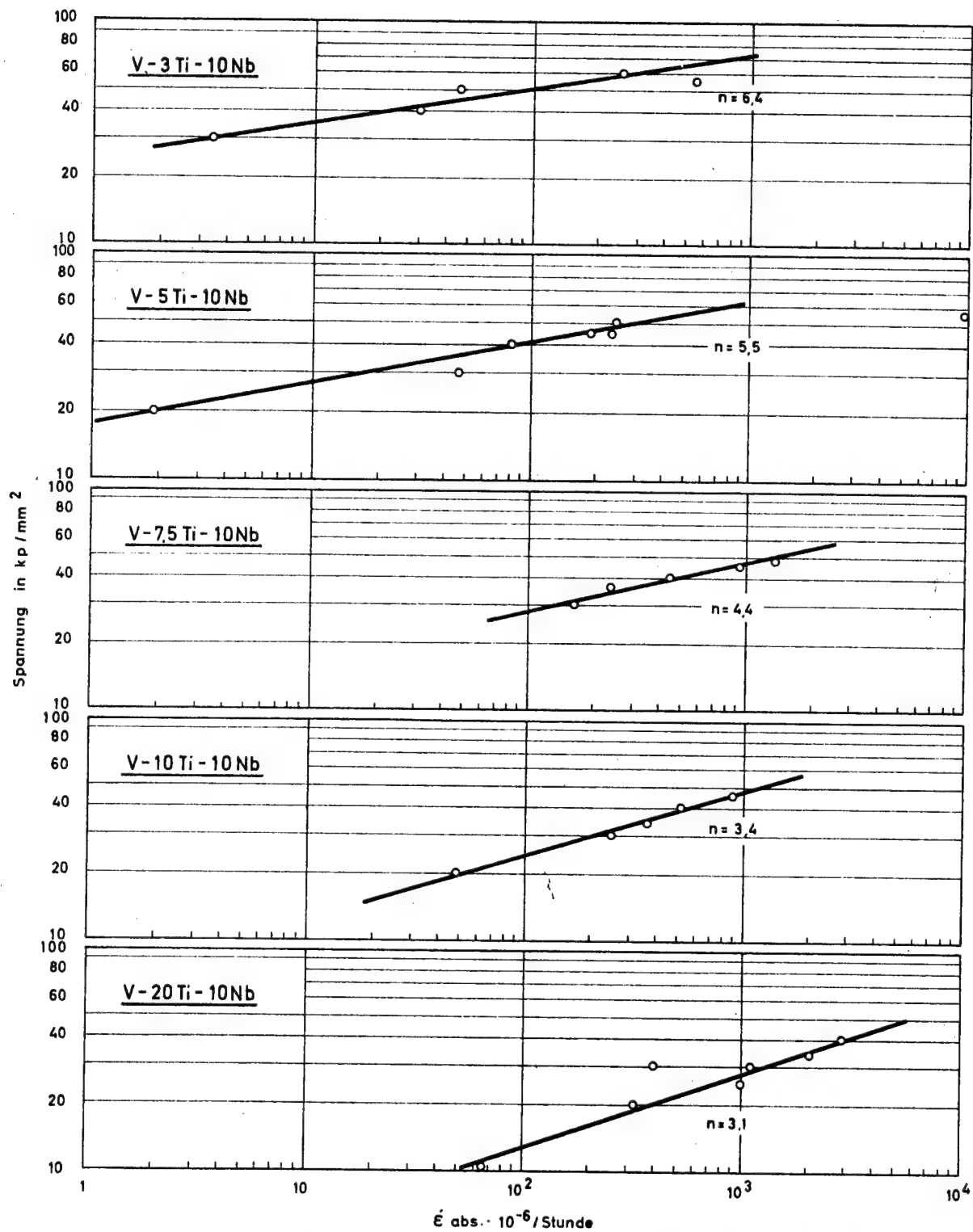
Bild 21: Abhängigkeit der sekundären Kriechgeschwindigkeit von der Spannung bei 650°C P ≈ 1 · 10⁻⁵ Torr

Figure axes and labels:
- Y-axis: Spannung in kp/mm²
- X-axis: É abs. · 10⁻⁶/Stunde

Panels:
- V-3 Ti-10 Nb, n = 6,4
- V-5 Ti-10 Nb, n = 5,5
- V-7,5 Ti-10 Nb, n = 4,4
- V-10 Ti-10 Nb, n = 3,4
- V-20 Ti-10 Nb, n = 3,1
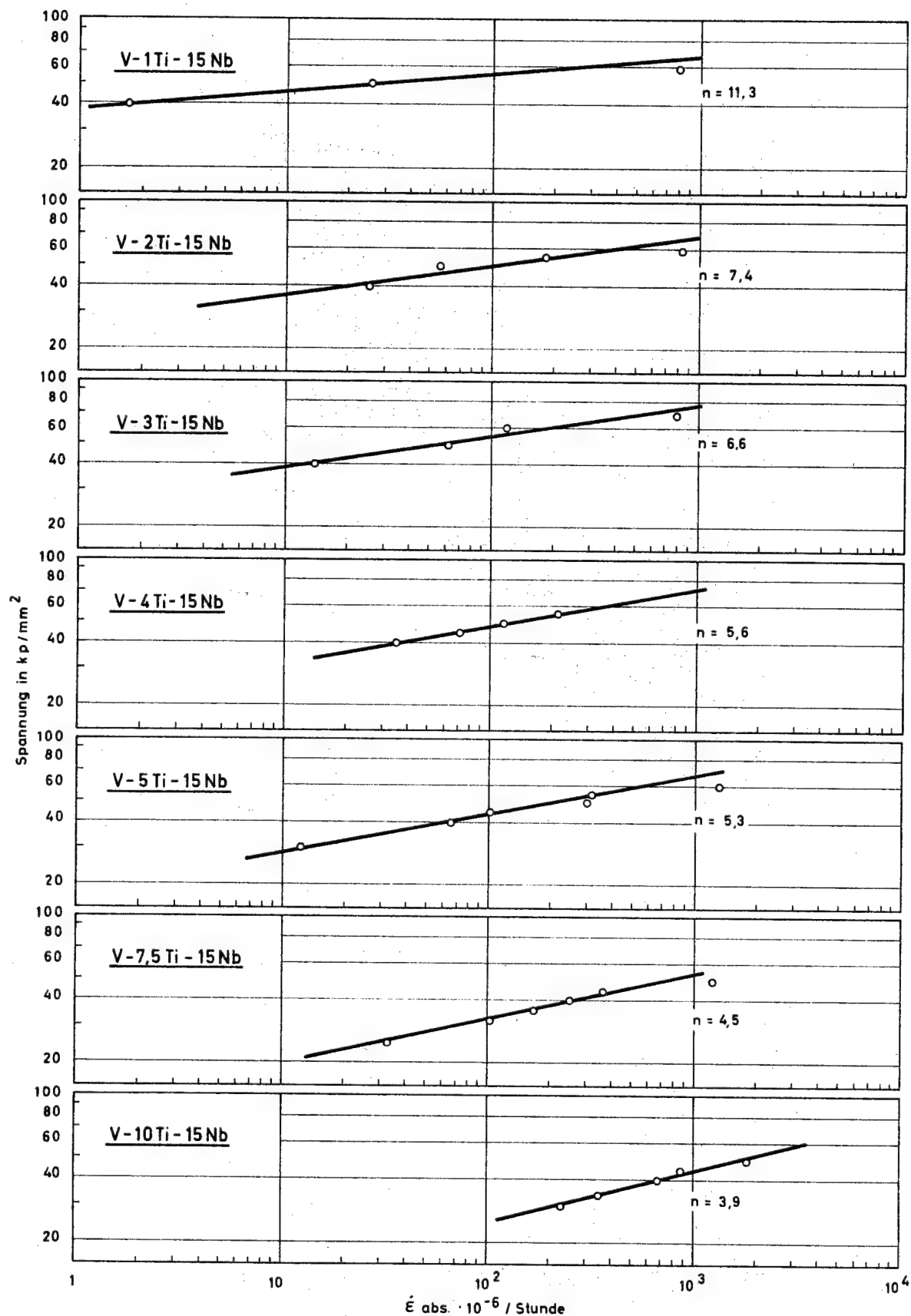
Bild 22 : Abhängigkeit der sekundären Kriechgeschwindigkeit von der Spannung bei 650°C
P ≈ 1·10⁻⁵ Torr
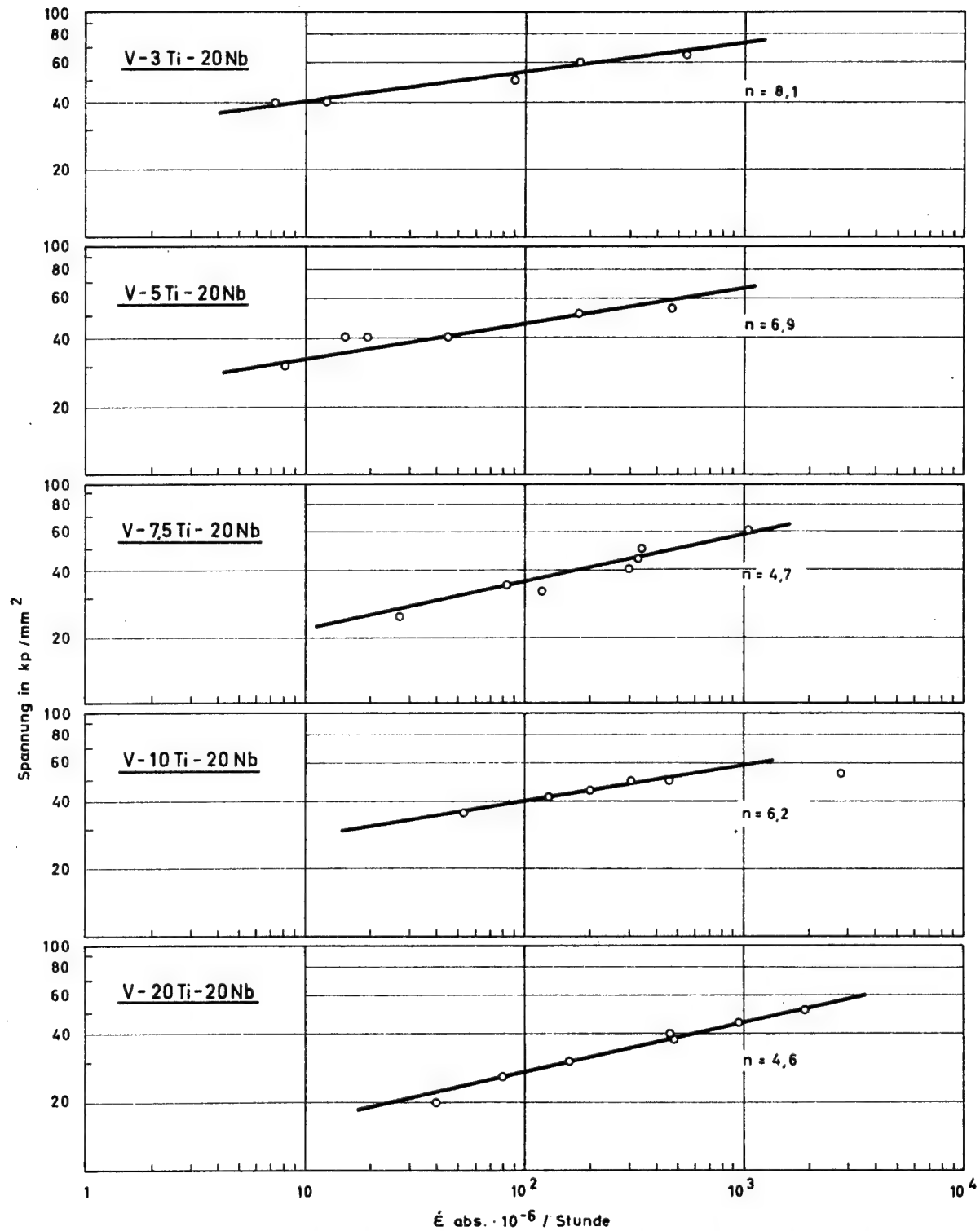
Bild *23*: Abhängigkeit der sekundären Kriechgeschwindigkeit von der Spannung bei 650 °C
$P \approx 1 \cdot 10^{-5}$ Torr
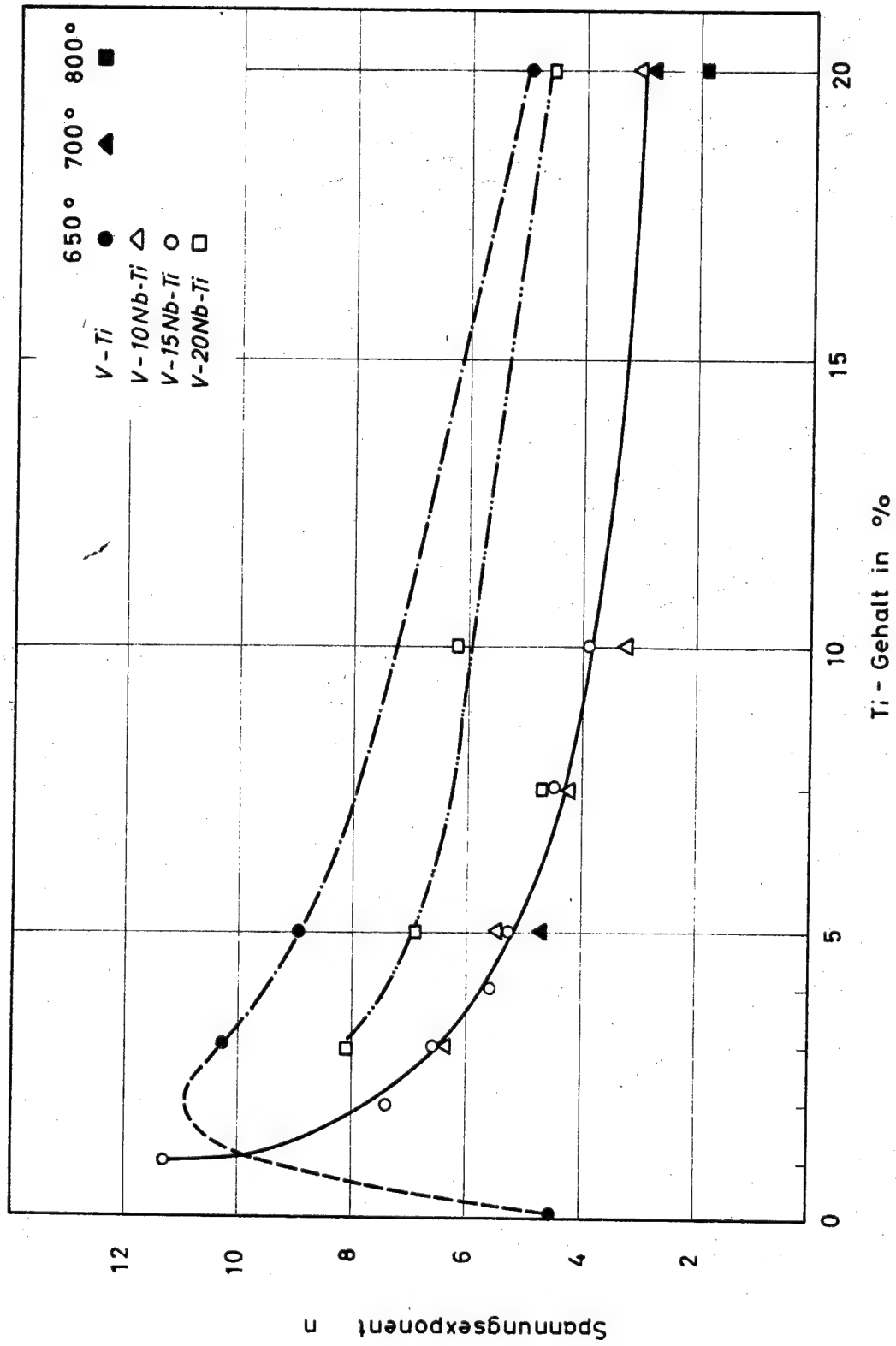
Bild 24 : Einfluß des Ti - Gehaltes auf den Spannungsexponenten binärer und ternärer Vanadin - Legierungen
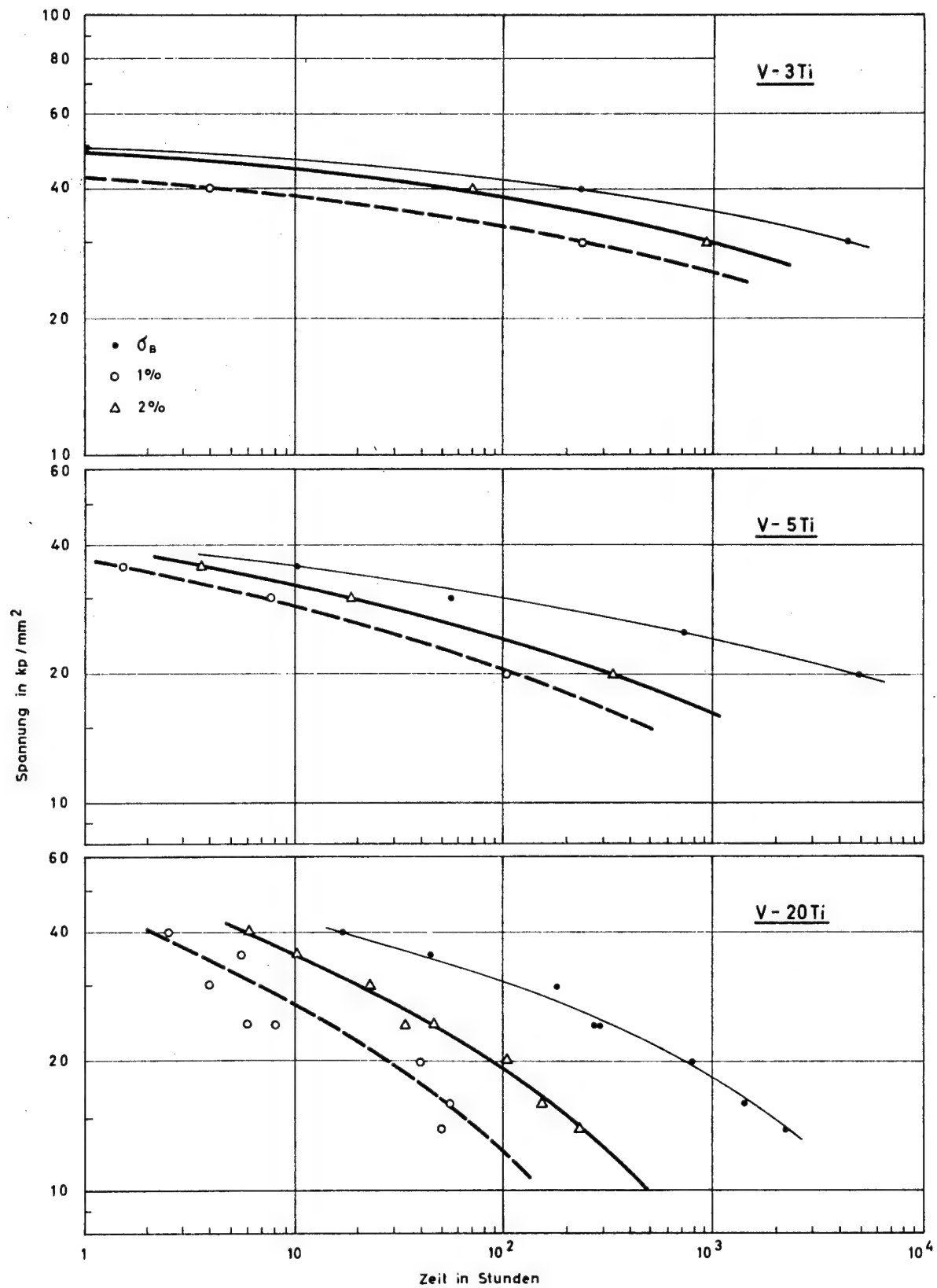
Bild *25* :   Zeit - Dehngrenzen in Abhängigkeit von der Spannung   T = 650 °C
P ≈ 1 · 10⁻⁵ Torr

V - 3 Ti - 10 Nb

V - 5 Ti - 10 Nb

V - 7,5 Ti - 10 Nb

V - 10 Ti - 10 Nb

V - 20 Ti - 10 Nb

Spannung in kp/mm²

Zeit in Stunden
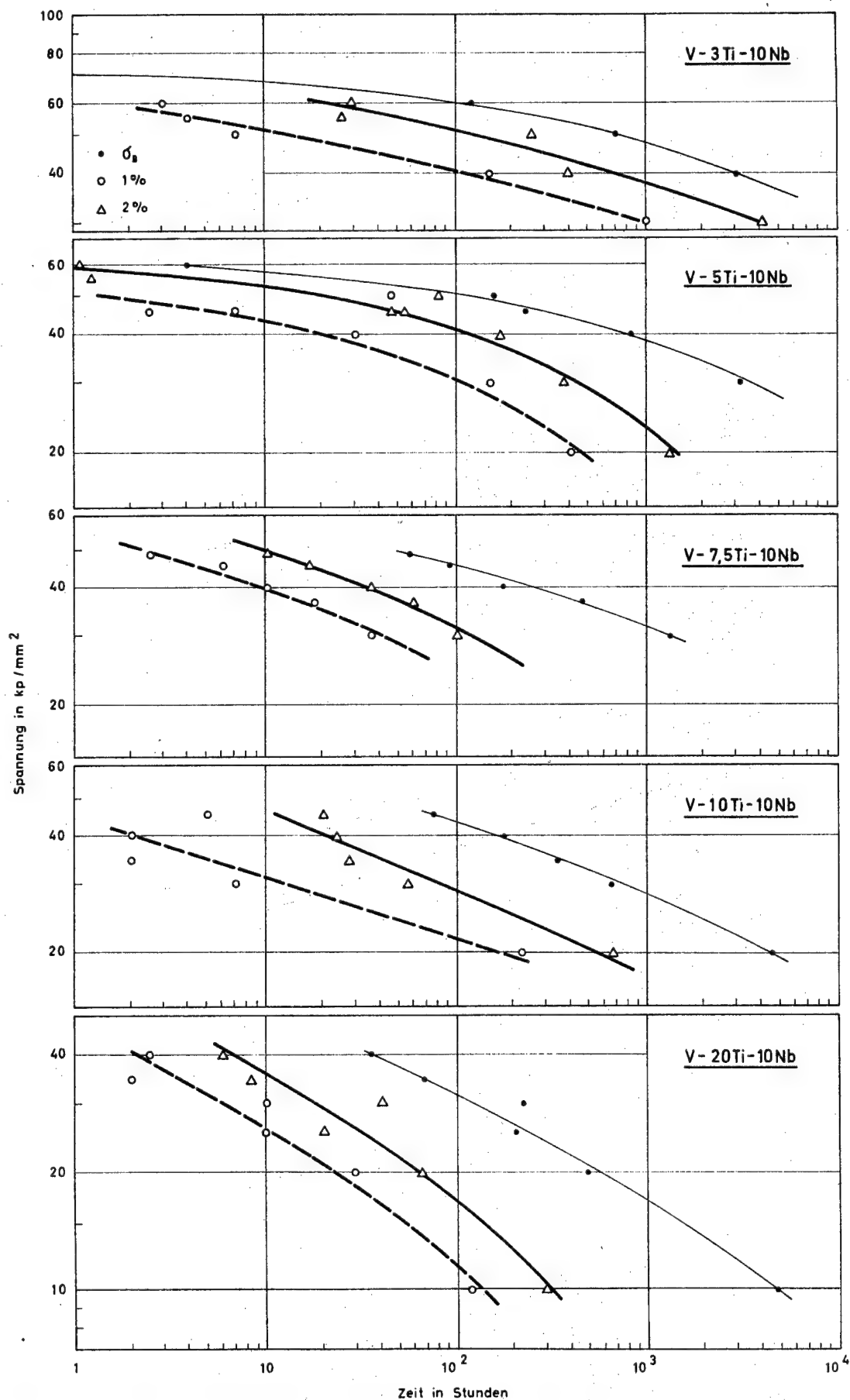
Bild 26 :   Zeit - Dehngrenzen in Abhängigkeit von der Spannung   T = 650 °C
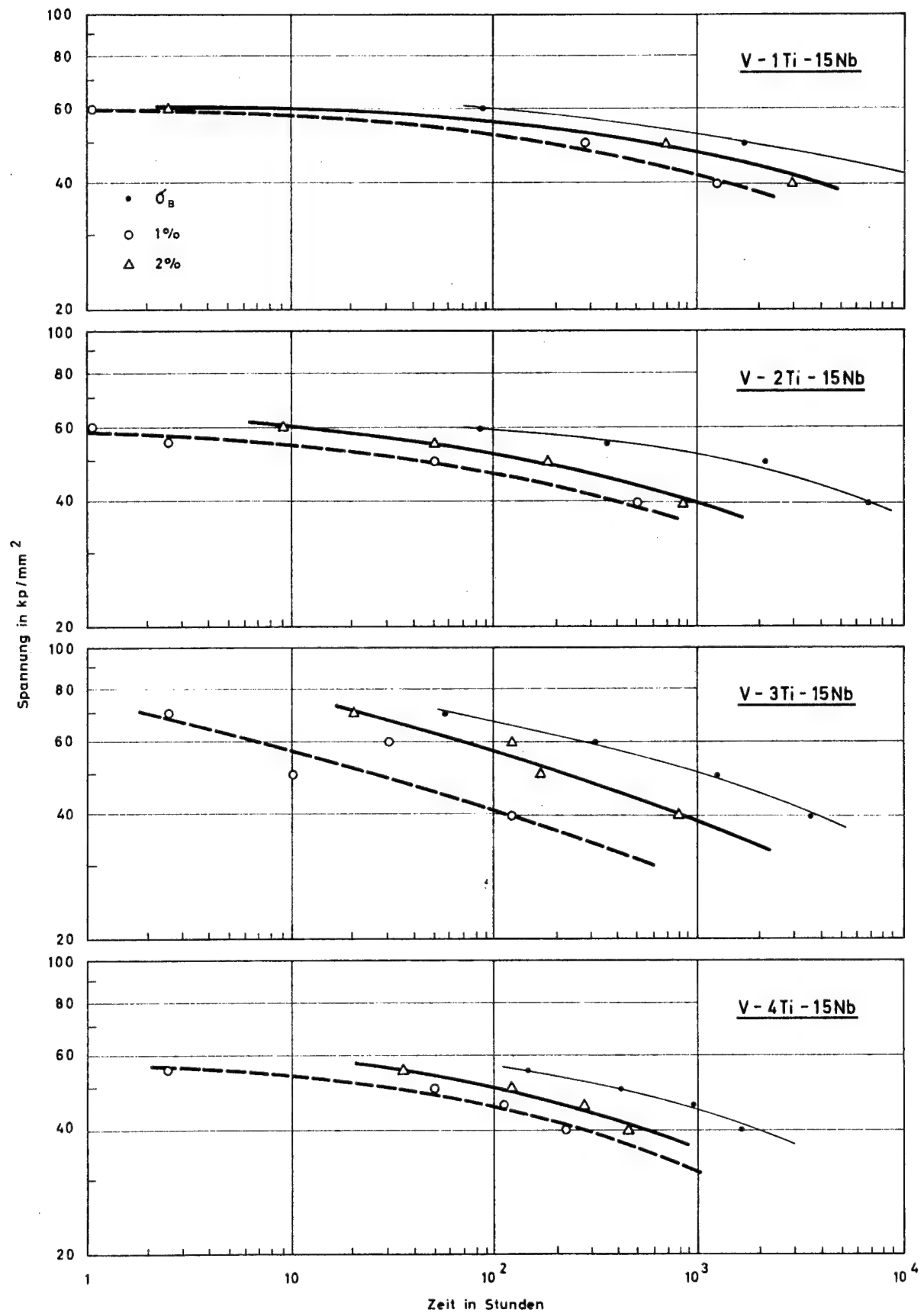
$P \approx 1 \cdot 10^{-5}$ Torr

Bild *27* :   Zeit – Dehngrenzen in Abhängigkeit von der Spannung   T = 650 °C
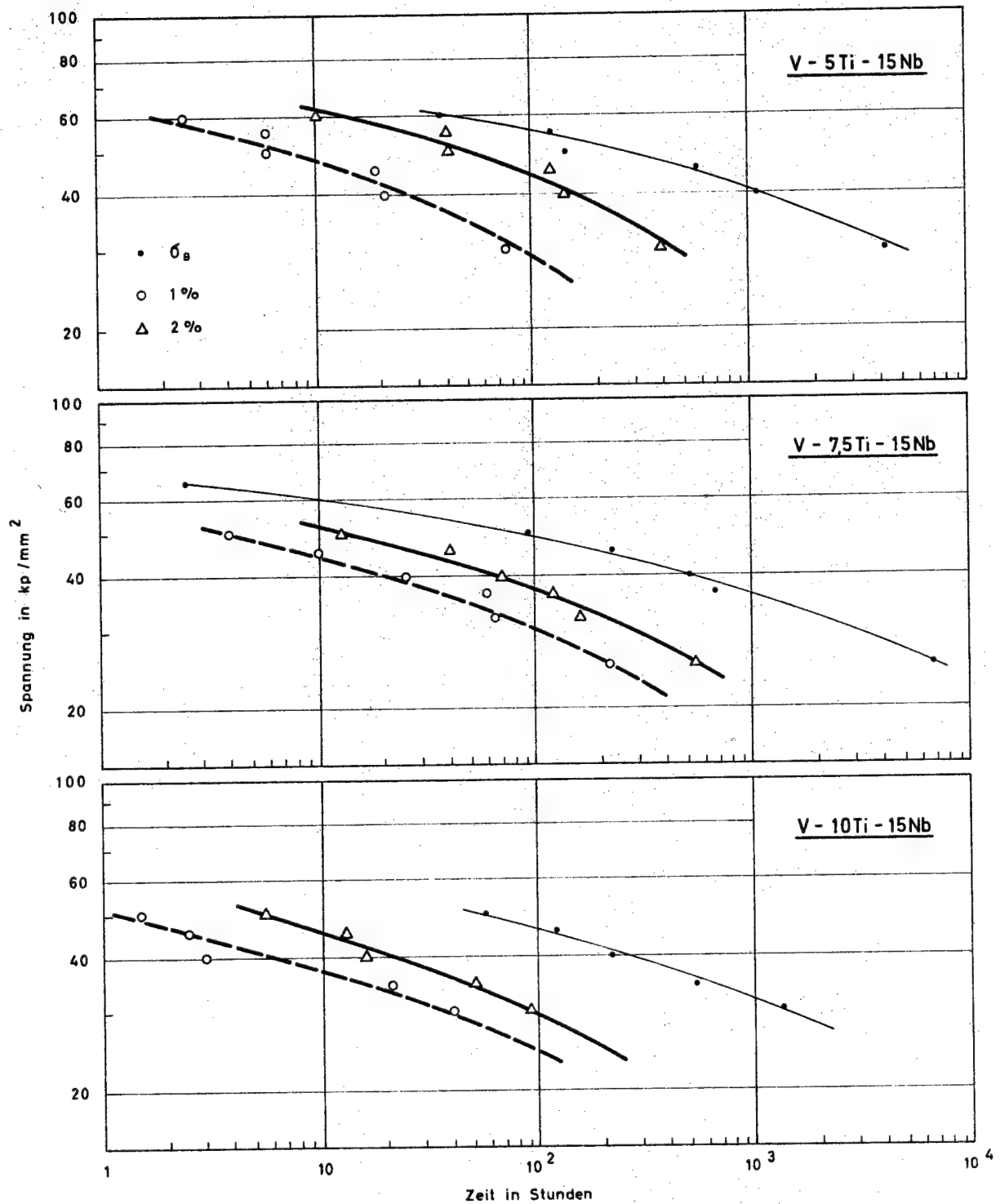
P ≈ $1 \cdot 10^{-5}$ Torr

Bild 28 :   Zeit -Dehngrenzen in Abhängigkeit von der Spannung   T = 650 °C
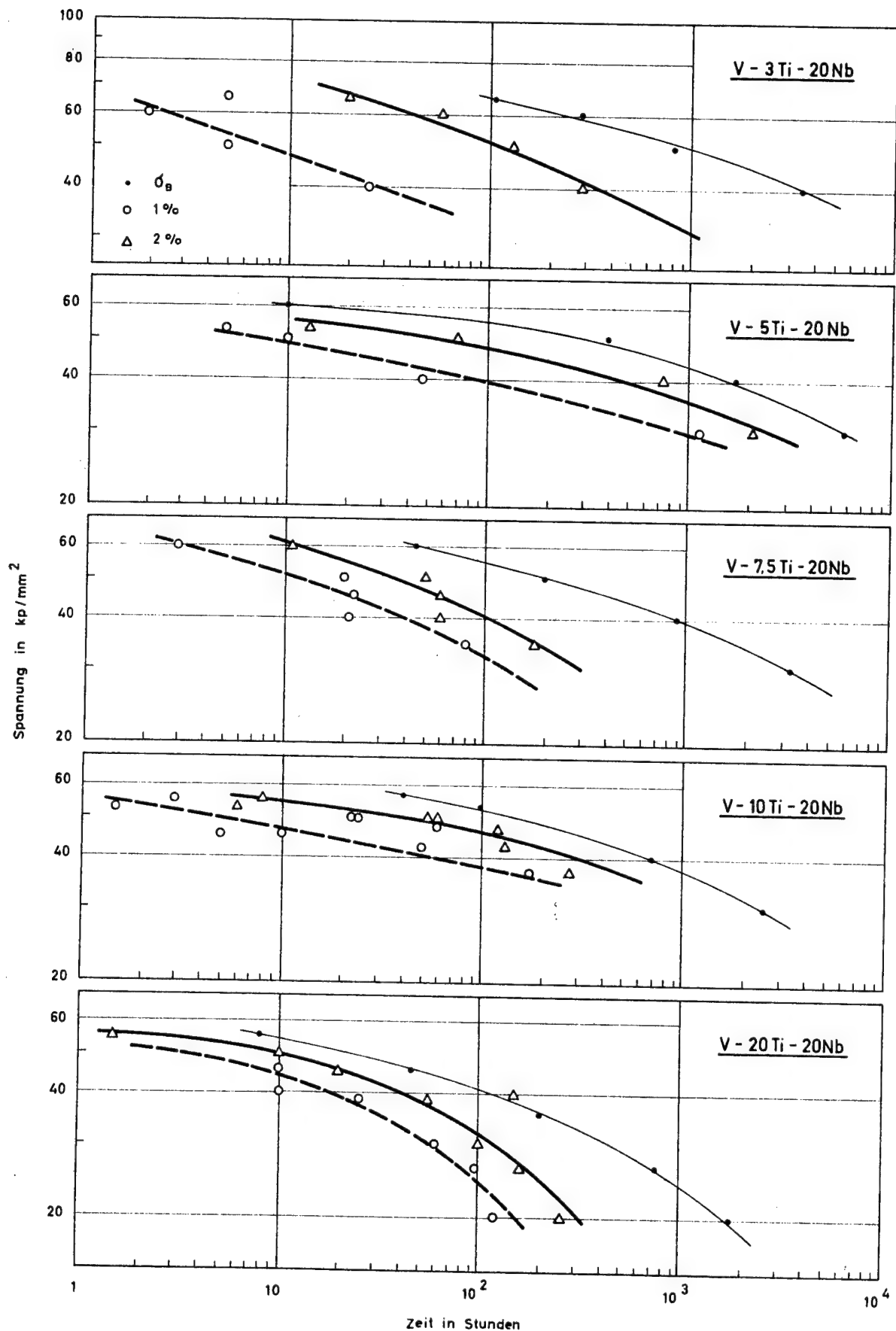
$P \approx 1 \cdot 10^{-5}$ Torr

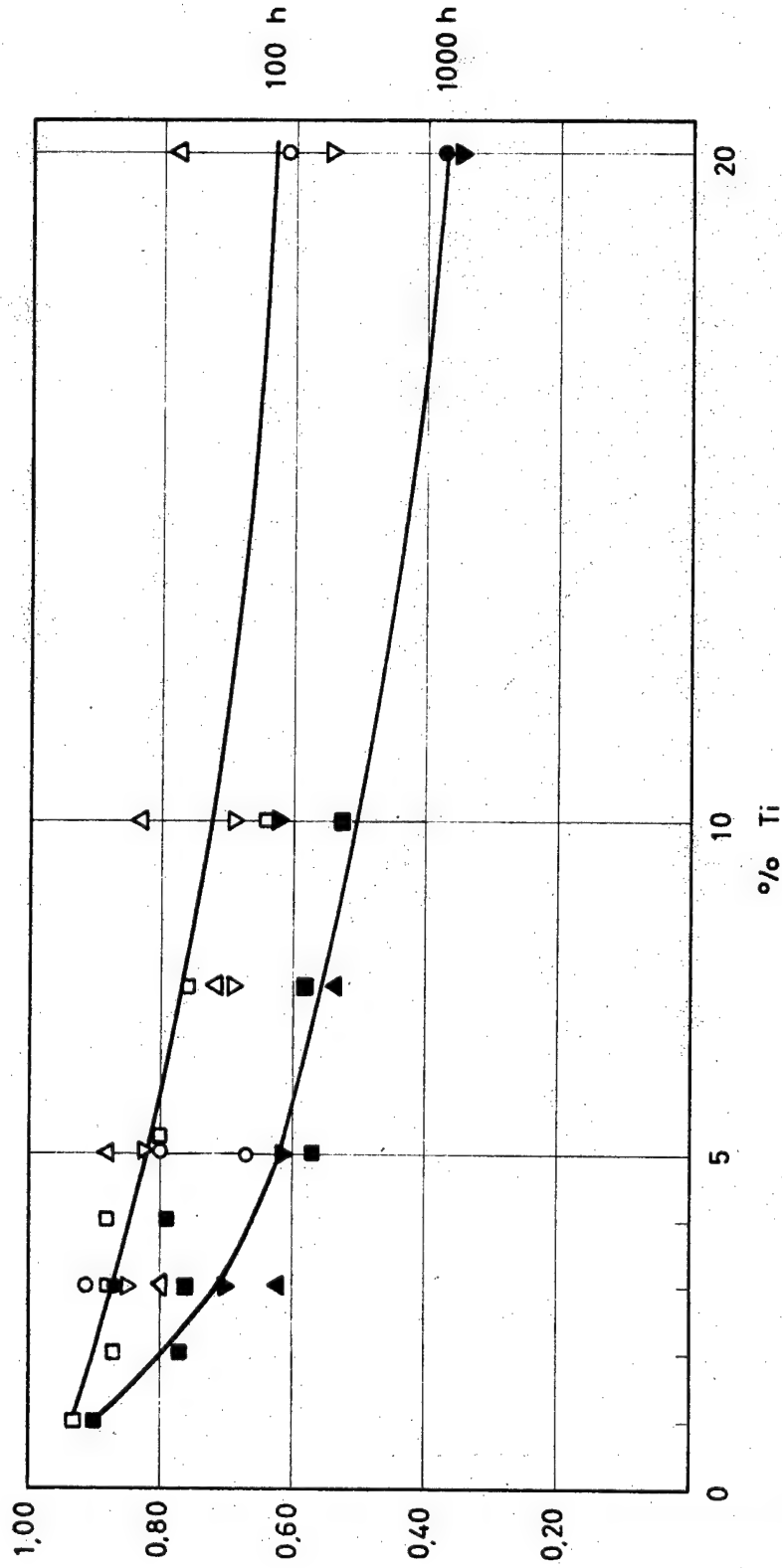Bild 29 : Zeit - Dehngrenzen in Abhängigkeit von der Spannung T = 650 °C
P ≈ 1·10⁻⁵ Torr

Bild 30: Verhältnis Zeitdehngrenze zu Zeitstandfestigkeit in Abhängigkeit vom
Ti – Gehalt für 2% Dehnung bei 100ʰ bzw. 1000ʰ Standzeit

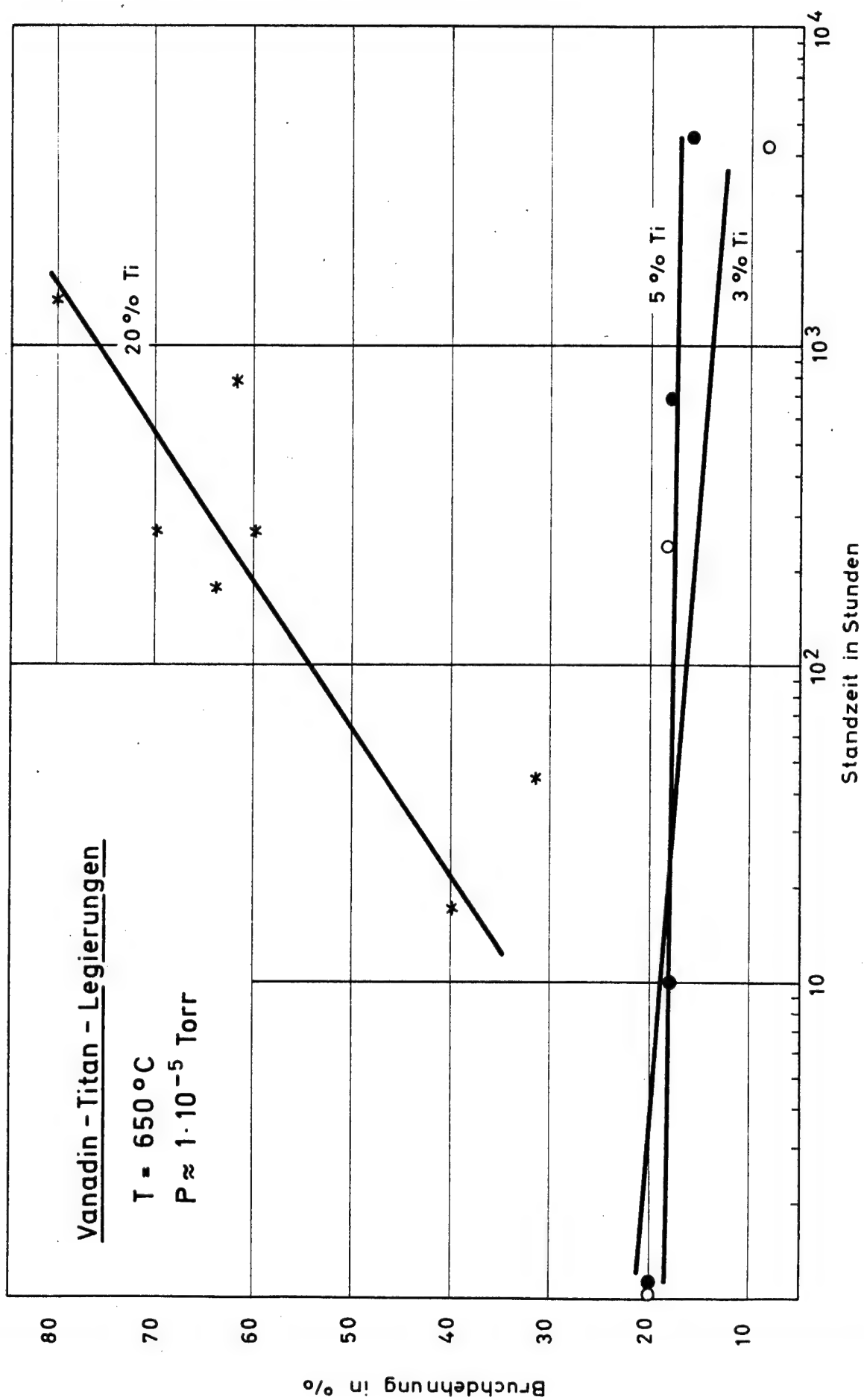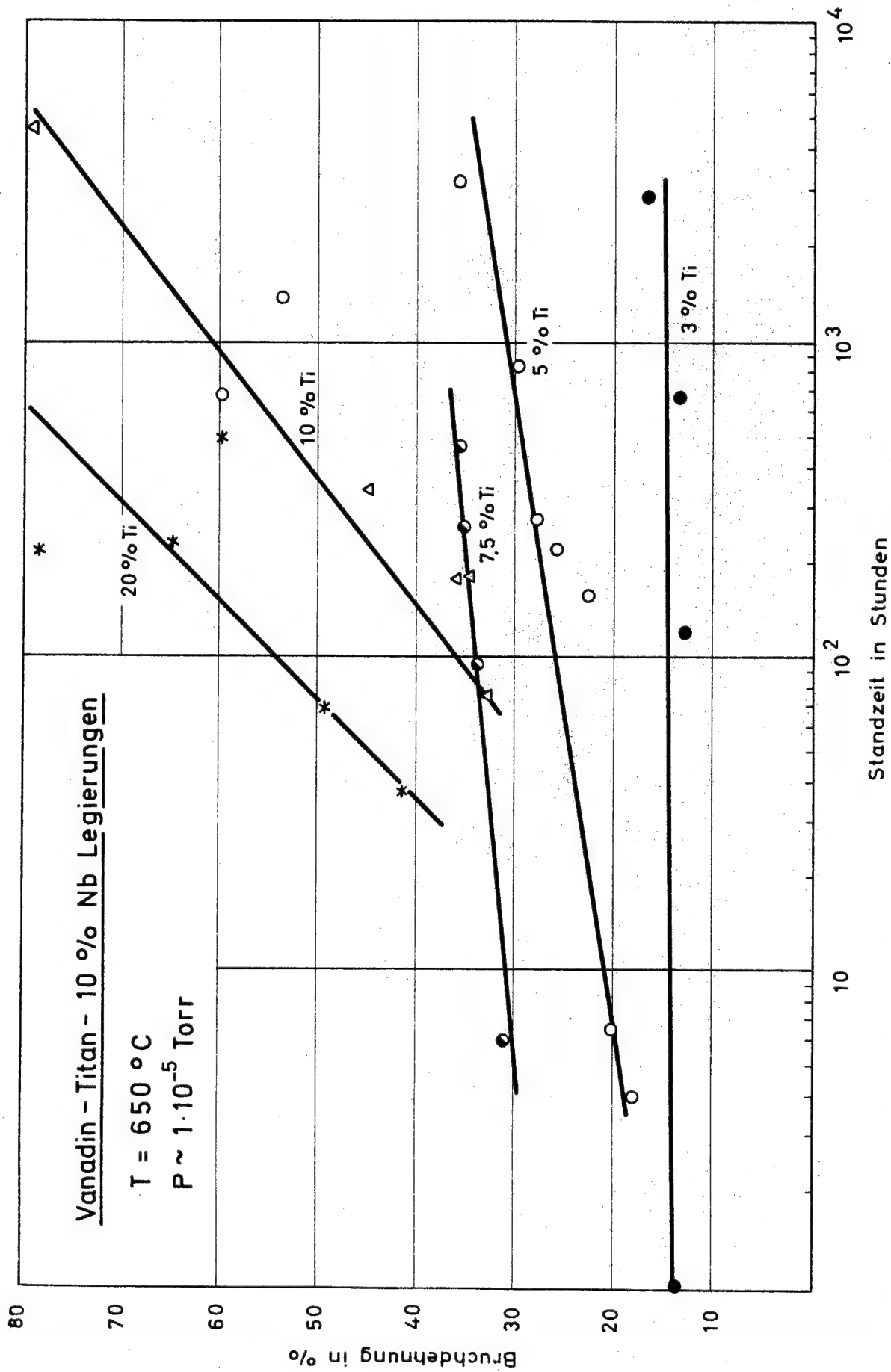Bild 31 :   Bruchdehnung in Abhängigkeit von der Standzeit bei verschiedenen Ti – Gehalten

Vanadin-Titan-10 % Nb Legierungen

$T = 650°C$
$P \approx 1 \cdot 10^{-5}$ Torr

Bruchdehnung in %

20 % Ti

10 % Ti

7,5 % Ti

5 % Ti

3 % Ti

Standzeit in Stunden

Bild 32 : Bruchdehnung in Abhängigkeit von der Standzeit bei verschiedenen Ti-Gehalten

Bruchgefüge
===========

V-2Ti-15Nb

$T = 650^{\circ}$

$\sigma = 40 \text{ kp/mm}^2$

$t_B = 6328 \text{ Std.}$

$HV_{30} = 227\text{-}235$

V-5Ti

$T = 700^{\circ}C$

$\sigma = 10 \text{ kp/mm}^2$

$t_B = 6630 \text{ Std.}$

$HV_{30} = 138\text{-}145$

x 100

Bruchgefüge
==========

V-3Ti-20Nb

$T = 850^o$

$\sigma = 6 \text{ kp/mm}^2$

$t_B = 2078 \text{ Std.}$

$HV_{30} = 215\text{-}244$

V-3Ti-15Nb

$T = 920^o$

$\sigma = 4 \text{ kp/mm}^2$

$t_B = 188 \text{ Std.}$

$HV_{30} = 194\text{-}198$
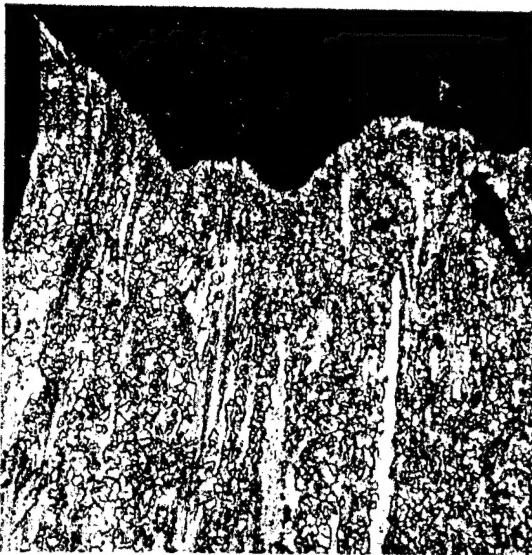
x 100

V-3Ti-20Nb

T   = 850°C

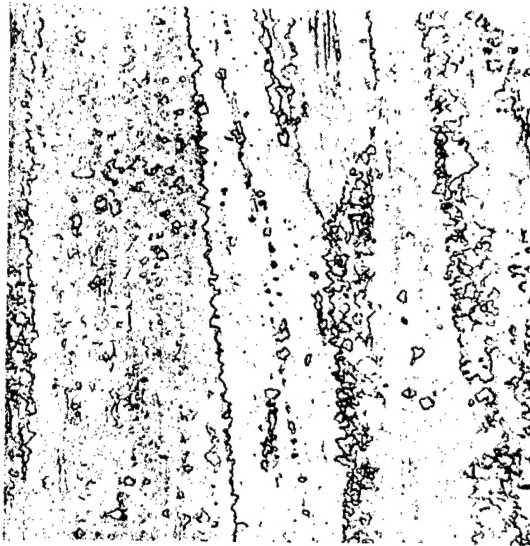σ   = 6 kp/mm^2

t_B = 2078 Std.

δ_{sd} = 123%

Bruchzone

HV_{30} = 215-244

Meßlänge

Kopf

These photographs reduced 10% in printing process.

HV_{30} = 210-224

x 100

Bild 36 : Sekundäre Kriechgeschwindigkeit in Abhängigkeit von der Standzeit